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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

1819. **Gas chromatography 1960. Proceedings of e third symposium, Edinburgh, June, 1960.** ganised by the Society for Analytical Chemistry d the Gas Chromatography Discussion Group of e Hydrocarbon Research Group of the Institute Petroleum. Publ. Butterworths, London, 1960. 6 pp. The following papers are included. **vestigation of the hydrogen flame ionisation tector,** L. Ongkiehong, pp. 1–15. **Argon detectors,** E. Lovelock, pp. 16–29. **Comparative data on o ionisation detectors,** R. D. Condon, P. R. holly and W. Averill, pp. 30–45. **An examination the flame ionisation detector using a diffusion ution apparatus,** D. H. Desty, C. J. Geach and Goldup, pp. 46–64. **Comparison of integral and ferential ionisation detectors for gas chromato-aphy,** S. Matoušek, pp. 65–80. **Gas analysis by e use of the discharge tube,** B. Riley, pp. 81–87. **pour flow chromatography,** J. Boeke, pp. 88–103. **new time - voltage integrator and further auto-ation of gas-chromatographic analysis,** I. Halász d W. Schneider, pp. 104–116. **Cathode-ray esentation of chromatograms,** R. P. W. Scott and A. Cumming, pp. 117–128. **Some factors affecting lumn efficiency and resolution of nylon capillary lumns,** R. P. W. Scott and G. S. F. Hazeldean, . 144–161. **Coated capillary columns—an in-stigation of operating conditions,** D. H. Desty and Goldup, pp. 162–183. **An approach to higher eeds in gas - liquid chromatography,** J. H. Purnell d C. P. Quinn, pp. 184–198. **Temperature nitations of stationary phases,** W. Gerrard, S. J. awkes and E. F. Mooney, pp. 199–210. **Structural alysis of hydrocarbons by capillary gas chromato-aphy in conjunction with the methylene insertion action,** M. C. Simmons, D. B. Richardson and I. voretzky, pp. 211–223. **Improvements in the ficiency of large diameter gas - liquid chromato-aphy columns,** F. H. Huyten, W. van Beersum d G. W. A. Rijnders, pp. 224–241. **Purification d identification of the components of complex ganic materials,** T. Johns, pp. 242–250. **Deter-ination and use of the specific retention volumes benzene and cyclohexane in dinonyl phthalate,** R. Adlard, M. A. Khan and B. T. Whitham, . 251–272. **The use of some complexes of the ansition metals as column liquids in gas chromato-aphy,** G. P. Cartoni, R. S. Lowrie, C. S. G. Phillips d L. M. Venanzi, pp. 273–283. **Retention times d molecular shape: the use of tri-o-thymotide in olumn liquids,** A. O. S. Maczek and C. S. G. Phillips, . 284–288. **Some developments in gas chromato-aphy in the USSR,** A. A. Zhukhovitsky, pp. 293–00. **Gas - liquid partition chromatography for paration of metal halides,** R. A. Keller and H. reiser, pp. 301–307. **The gas-chromatographic analysis of inorganic halogen compounds on capillary columns,** T. R. Phillips and D. R. Owens, pp. 308–320. **Qualitative and quantitative analysis, by gas - solid chromatography, of mixtures containing nitrogen oxides,** L. Marvillet and J. Tranchant, pp. 321–330. **A gas-chromatographic apparatus for in-line analysis of corrosive inorganic gases,** G. Iveson and A. G. Hamlin, pp. 333–343. **The application of gas chromatography to anaesthetic research,** D. W. Hill, pp. 344–353. **Analysis of full boiling range gasolines by chromatographic methods,** R. V. Blundell, S. T. Griffiths and R. R. Wilson, pp. 360–371. **A new approach to the study and assessment of medicinal white oil stability,** C. G. Scott, pp. 372–386. **Identification of organic substances by the gas-chromatographic analysis of their pyrolysis products,** J. Janák, pp. 387–400. **The gas chromatography of ethylenically unsaturated compounds with particular reference to esters,** C. E. R. Jones, pp. 401–411. **Fuel gas analysis: an apparatus incorporating a multi-cell thermal conductivity detector,** G. R. Boreham and F. A. Marhoff, pp. 412–422.

1820. **Radioactivation analysis. Proceedings, Radioactivation Analysis Symposium, Vienna, June 1959.** Organised by the International Atomic Energy Agency, Joint Commission on Applied Radioactivity (I.C.S.U.). Publ. Butterworths, London, 1960. 141 pp. The following papers are included, in English unless otherwise stated. **Radioactivation analysis in a nuclear reactor,** G. B. Cook, pp. 15–30. **Spectrometer for the determination of positron and gamma-ray cascade-emitting nuclides,** K. Ljunggren, pp. 31–34. **Neutron activation applied to geochemistry,** W. Herr, pp. 35–52. **Radioactivation analysis in the service of industry,** P. Leveque, pp. 53–60 (in French). **Experience in the U.S.A. on the use of radioactivation analysis,** G. W. Leddicotte, pp. 61–80. **Radioactivation analysis in biochemistry and medicine,** J. M. A. Lenihan, pp. 81–97. **Activation analysis of minor constituents in high-alloy steels,** J. Hoste, pp. 99–110. **Systematic analysis of zone-refined aluminium and iron after irradiation in the atomic pile,** P. Albert, pp. 111–119 (in French). **Determination of trace-element diffusion in quartz and in germanium,** G. Leliaert, pp. 121–126. **Method for the detection of mercury by radioactivation analysis,** K. Ljunggren and T. Westermark, pp. 127–133. **Determination of tantalum in high-tantalum ferro-alloys,** D. Gibbons and H. Simpson, pp. 135–137. **Intermetallic diffusion in gold - lead systems,** A. Ascoli and E. Germagnoli, pp. 139–141.

1821. **A general formula for the relationship between reagent addition and potential change in quantitative analysis.** F. L. Hahn (Angel Urraza 718–303, Mexico City 12, D.F., Mexico). *Z. anal. Chem.*, 1960, **177** (2), 113–121 (in German).—A procedure is described that enables the exact end-point in a potentiometric titration to be calculated. The formula has been applied to

acid - base, oxidation - reduction and precipitation titrations. T. R. ANDREW

1822. **Analysis for industry.** A. M. G. Macdonald. *Ind. Chem.*, 1960, **36**, 512–514.—The use of *N*-benzoyl-*N*-phenylhydroxylamine as an analytical reagent is discussed. (14 references.)
S.C.I. ABSTR.

1823. **Butyl rhodamine—a new reagent for photometric determinations, for extractions, precipitation and co-precipitation of elements.** V. I. Kuznetsov and L. I. Bol'shakova (V.I. Vernadsky Inst. of Geochem. and Anal. Chem., Acad. of Sci., USSR, Moscow). *Zhur. Anal. Khim.*, 1960, **15** (5), 523–527.—The butyl ether of rhodamine B (C.I. Basic Violet 10) (**I**) (prepared by heating the anhyd. sodium salt of **I** with butyl bromide under reflux at 120° for 10 to 12 hr.) is recommended for the separation and determination of elements that form hydrophilic complex anions in acid soln. Compared with **I** and other reagents it can be used over a wider range of acidity for the extraction of Tl, and gives higher recoveries of Pu^{IV} by co-pptn. from HNO_3 - NH_4NO_3. J. W. PRICE

See also Abstracts—**1845, 1872,** Dalzin as reagent. **1871,** 1,2-Dimorpholinoethane as reagent. **1904,** Complexometric indicator for Th. **1949,** Use of 3-hydroxy-3-phenyl-1-*p*-sulphophenyltriazen for F^-.

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

1824. **Influence of the solvent on the composition of extracted inorganic compounds.** H. Specker, E. Jackwerth and G. Hövermann (Inst. f. Spektrochem. u. angew. Spektroskopie, Dortmund-Aplerbeck). *Z. anal. Chem.*, 1960, **177** (1), 10–14 (in German).—The composition of the compound extracted from aq. soln. of $Fe(SCN)_3$, $Zn(SCN)_2$, BiI_3 and CdI_2 depends on the nature of the solvent. The more active solvents (*i.e.*, those of high partition coeff.) will replace one or more ligands in the solute, but the sum of the thiocyanate or halide and the solvent ligands remains constant.
J. H. WATON

1825. **Density - composition tables for aqueous solutions of sodium chloride and calcium chloride.** British Standards Institution (2 Park Street, London, W.1). B.S. 823:1960. 32 pp.—The text of B.S. 823:1938 has been revised to accord with the hydrometer standard B.S. 718.

1826. **New method of determining gases in metals.** Yu. A. Klyachko and O. D. Larina (I.P. Bardin Central Sci. Res. Inst. of Ferrous Metallurgy). *Zavod. Lab.*, 1960, **26** (9), 1047–1051.—The method is based on the analysis of gases set free by anodic dissolution. Electrolysis is carried out in a special vessel with anode and cathode compartments, separated by a filter-paper membrane, and a form of gas analyser. The electrolyte for determining H in steel contains 150 g of NaCl and 25 g of K Na tartrate per litre, the current density is 0·6 amp. per sq. cm, and the potential, temp., pH and duration of electrolysis are varied as required. The method is considered applicable to the determination of N as well as H, but not of O unless anodic dissolution is carried out in a non-aqueous medium, *e.g.*, with samples of titanium, zirconium and vanadium. G. S. SMITH

1827. **Polarographic determination of copper, zinc and chromium in a catalyst.** E. T. Saranchina and M. G. Dzyubenko (Lisichansk Chem. Combine). *Zavod. Lab.*, 1960, **26** (9), 1077–1078.—The sample (0·1 to 0·15 g) is dissolved in 2 to 3 ml of 35% HNO_3 and the soln. is evaporated to dryness. The residue is dissolved by addition of 10 ml of hot water and 0·5 ml of 35% HNO_3. The filtered soln. is diluted to 100 ml. A 1-ml aliquot is treated with 2 ml of N-NaOH and 1 ml of 0·01% gelatin soln. O is removed by passage of H and the polarogram covering the waves of Cu ($E_{\frac{1}{2}} = -0{\cdot}3$ V *vs.* the mercury anode), Cr ($E_{\frac{1}{2}} = -0{\cdot}8$ V) and Zn ($E_{\frac{1}{2}} = -1{\cdot}35$ V) is recorded. The wave heights are approportional to concn. G. S. SMITH

1828. **Quantitative isolation of small amounts of electromagnetically enriched isotopes of silver, zinc and cadmium.** U. Drehmann and K. Köning (Kernphysik. Inst., Zeuthen, Germany). *J. prakt. Chem.*, 1960, **11** (3–4), 121–124.—In the isolation of the enriched isotopes ^{107}Ag, ^{109}Ag, ^{64}Zn, ^{70}Zn and ^{110}Cd, the products from the isotope separator are contaminated with much of the material of the container, which must be made of a good heat conductor such as copper, graphite, silver or aluminium. Chemical methods of purification are described. Silver was separated as AgSCN after treating the bombarded aluminium containers with 30% HNO_3, when much Al goes into solution with the Ag. The ^{64}Zn and ^{70}Zn were separated electrolytically from considerable amounts of Cu, or by extraction of the Zn as $Zn(SCN)_2$ with isobutyl methyl ketone. Separation of ^{110}Cd from a large excess of Cu was effected by double pptn. as sulphide from a soln. of the cyano-complexes.
N. E.

1829. **Acid stability and the analysis of nitrides of titanium, zirconium, niobium and tantalum.** O. I. Popova and G. T. Kabannik. *Zhur. Neorg. Khim.*, 1960, **5** (4), 930–934; *Ref. Zhur., Khim.* 1960, (17), Abstr. No. 69,239.—To determine the metals, the nitrides of Zr or Nb (0·1 g) were decomposed with conc. H_2SO_4, pptd. with cupferron and ignited to Nb_2O_5 or ZrO_2. Nitrides of Ta or Ti were dissolved in HNO_3 - HF and evaporated to the appearance of H_2SO_4 fumes; Ti was pptd. with cupferron and Ta with aq. NH_3 soln., and the ppt. was ignited to Ta_2O_5 or TiO_2. For N, the sample (0·1 g) were dissolved in conc. H_2SO_4 (10 ml) (for Zr or Nb) or in a mixture of conc. H_2SO_4 (10 ml) with K_2SO_4 (5 to 7 g) and treated by the Kjeldahl procedure. Determination of the metals in nitrides can also be carried out by simple ignition of the original samples (0·1 g) at 1000°; nitrides are converted into oxides. K. R. COOK

1830. **Spectrographic determination of traces of chromium, vanadium and nickel in refined brine for the mercury electrolysis process.** Hiroshi Suzuki, Eizo Yasui and Kumao Ohashi (Res. Inst., Tokuyama Gosei Chem. Ind., Showa-cho, Minato-ku, Nagoya). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1959, **62** (5), 617–621.—Three drops of an acid soln. (containing $\lt 1$ μg of Cr or V and $\lt 2{\cdot}5$ μg of Ni pe

)0 ml) are evaporated to dryness on a carbon ectrode (diameter 6 mm, length 20 mm) and an ermittent arc (4·7 amp.; terminal voltage, 210 V; ity factor, 1/3; period, 1 sec.; preliminary scharge, 10 sec.; exposure, 2 min.) is struck etween this and a second carbon electrode (gap mm); K and Na increase the sensitivity, whereas H_4^+ decrease it. Calcium and Mg interfere, and ust be removed. Iron is added as internal stand-rd; the line pairs used are Cr 2677·16 *vs.* Fe 2689·21; 3110·71 *vs.* Fe 3116·63; and Ni 3414·77 *vs.* e 3399·34. *Procedure for the preparation of the mple soln.*:—Boil the soln. (100 ml) with HCl :1) (2 ml), add Fe^{II} soln. (5 mg of Fe), NH_4Cl g) and conc. HNO_3 (5 drops), precipitate $Fe(OH)_3$ ith aq. NH_3 soln., filter, wash the ppt. with 0·5N-aOH (30 ml) and hot water (5 ml), dissolve it in Cl (1:1) (10 ml) and water (20 ml), evaporate to ryness, moisten the residue and neutralise the xcess of HCl with 0·05N-aq. NH_3. K. Saito

1831. **Determination of small amounts of hydrogen n helium.** A. A. Datskevich, L. P. Zhigacheva, . V. Krasnova, M. D. Lapitskaya, A. G. Latukhova nd M. B. Moshinskaya (Engng Bureau of Auto-natics and Telemechanics). *Zavod. Lab.*, 1960, **26** 9), 1082–1083.—An automatic chromatographic ethod, with two detectors, based on thermal-onductivity measurement for He and heat of ombustion for H, is briefly described.
G. S. Smith

1832. **Quantitative determination of water in eavy water and butanol by near-infra-red spectro-netry.** Kaoru Sakai, Fumio Mizuniwa, Tsurayuki Akamatsu, Shozo Matsumoto, Yasuo Kurihara and Masami Shimizu (Hitachi Res. Lab., Hitachi, baraki-ken). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1959, **62** (5), 632–635.—For the determination f water (<1·2%, w/w) in butanol (**I**), the absorp-ion at 1·92 μ is used, with **I** free from water as eference. The error is from 0·2 to 1%. For the letermination of water in heavy water, the absorp-ions at 1·18 μ and 1·45 μ are useful, although they hift with change in concn. of H_2O in ^2H_2O. The rror is $\simeq$0·25% for >20%, and $\simeq$0·6% for <20% f H_2O, respectively. K. Saito

1833. **Inorganic circular paper chromatography. V. Separation of alkali metals.** A. R. Vasudeva Murthy and V. A. Narayan (Dept. of Inorg. and Phys. Chem., Indian Inst. Sci., Bangalore). *J. Indian Inst. Sci.*, 1960, **42** (3), 51–55.—Lithium, sodium and potassium salts are separated and identified by adsorption indicators, the best being $AgNO_3$ with eosin in ethanol, or $AgNO_3$ with phenosafranine in acetone, applied by dipping. The best irrigating solvent for sharp separation is methanol - ethanol (1:1). Attempts to effect the separation of the chlorides, bromides and iodides of the same alkali metal proved unsuccessful with ethanol, methanol and butanol satd. with H_2O, owing to the closeness of the R_F values; with anions of the same cation, only LiI could be separated from the bromide and chloride on a larger paper with a longer irrigation time. Butanol satd. with 4N-acetic acid (*Anal. Abstr.*, 1956, **3**, 371) did not separate the halide ions. S.C.I. Abstr.

1834. **Sodium hydroxide (photographic grade).** British Standards Institution (2 Park Street, London, W.1). B.S. 3308:1960. 8 pp.—Tests for purity and a method of assay are given.

1835. **Radiometric determination of potassium in sodium metal, a ternary alloy, and electrolytes.** I. M. Korenman, F. R. Sheyanov, A. A. Tumanov, Z. I. Glazunova and O. I. Demin. *Trudy Khim. i Khim. Tekhnol.*, 1959, (1), 94–101; *Ref. Zhur., Khim.*, 1960, (17), Abstr. No. 69,131.—*Procedure*—Dissolve the benzene-washed, dried sample (0·5 to 0·8 g) in ethanol (10 ml), neutralise the soln. with conc. HCl (to phenolphthalein), cool, and dilute to 25 ml; acidify an aliquot (1 ml) with 2N-acetic acid (0·2 ml), add 5% $Co(NO_3)_2$ soln. (0·25 ml), containing ^{60}Co, and 50% $NaNO_2$ soln., set aside for 1 hr. and centrifuge. Wash the residue of $K_2Na[Co(NO_2)_6]$ with acetone, dissolve it by heating in 0·5N-HNO_3 and dilute the soln. to 0·5 ml. Determine the K content radiometrically in 0·25 ml of soln. The determination takes 2 to 3 hr. The method can be applied to the ternary alloy of potassium, sodium and lead. The composition of the residue in this case corresponds to $KPb[Co(NO_2)_6]$; pptn. is carried out at <20°. Potassium in electrolytes containing NaCl, KCl, NaF or Na_2CO_3 is determined by the natural radioactivity of K. The powdered salt is placed in a special cylindrical cell and the activity of ^{40}K is measured; calibration curves are constructed with a NaCl - KCl mixture. The determination takes 20 min. K. R. Cook

1836. **Flame-photometric determination of potassium in rubidium chloride.** B. D. Stepin and V. E. Plyushchev (M. V. Lomonosov Moscow Inst. of Fine Chem. Technol.). *Zhur. Anal. Khim.*, 1960, **15** (5), 556–560.—By using the internal standard method, amounts of K in the range 0·005 to 0·01% can be determined by comparing the intensities of the lines K 7665A and Rb 7800A. The concn. of the base soln. of RbCl was 4%. Sodium, Cs and Ca in the range 0·001 to 0·5% do not interfere, but Mg in concn. higher than 0·1% decreases the emission of the K line. Small amounts of Rb in potassium chloride can be determined similarly.
J. W. Price

1837. **Determination of potassium by its natural radioactivity.** I. M. Korenman and B. A. Nikolayev. *Trudy Khim. i Khim. Tekhnol.*, 1959, (1), 102–104; *Ref. Zhur., Khim.*, 1960, (17), Abstr. No. 69,130.—In the method previously described [*Ref. Zhur., Khim.*, 1956, (18), Abstr. No. 58,433], the amount of sample was decreased to 1 g; the sensitivity was $\simeq$ 1 count per min. with a 1% concn. of K in the sample. The method is not suitable for the determination of <5% of K. K. R. Cook

1838. **Determination of potassium in mica: a flame-photometric study.** S. Abbey and J. A. Maxwell (Anal. Chem. Sect., Petrological Sci. Div., Geological Survey of Canada, Ottawa). *Chem. Canada*, 1960, **12** (9), 37–41.—Pulverised mica is heated with HF - H_2SO_4. Excess of HF is removed, $MgSO_4$ is added in known excess, and excess of H_2SO_4 is removed by evaporation. From the ignited sulphates a neutral residue containing the alkali metals and Mg is obtained. Potassium is determined at 766, Rb at 780, Li at 671 and Na at 589 mμ in a spectrophotometer with flame attachment and photomultiplier. Adsorption of K by Al_2O_3 or Fe_2O_3 is prevented by addition of $MgSO_4$, but too much $MgSO_4$ depresses the K emission.
O. M. Whitton

1839. **Caesium in chondrite meteorites.** L. H. Ahrens, R. A. Edge and S. R. Taylor (Dept. of Chem., Univ. of Cape Town, S. Africa). *Geochim.*

et Cosmoch. Acta, 1960, **20** (3–4), 260–272.—As the concn. of Cs in chondrites is low ($\simeq$0·1 p.p.m.), the principal constituents (Si, Mg, Fe, Al, Ca and Na) have to be almost completely removed. A four-step scheme is used (after sample dissolution and removal of Si with HF)—(*i*) removal of Fe (as a chloro-complex) on an Amberlite IRA-400 anion-exchange column, (*ii*) removal of Al and some Mg and Ca through perchlorate decomposition and leaching with H_2O, (*iii*) removal of Na (first Dowex-50 cation-exchange column), and (*iv*) removal of remaining Mg (second Dowex-50 cation-exchange column). The K - Rb - Cs concentrate is then analysed spectrographically by using the line Cs 8521 A; K to Cs ratios are determined by using the additional line K 6911 A. Full details are given of the ion-exchange enrichment processes, which are also adapted for determinations of Cs in basic rocks. R. A. Howie

1840. **Analysis of oxide films on copper by coulometric reduction.** A. Rönnquist (Inst. Chem., Univ., Uppsala, Sweden). *Acta Chem. Scand.*, 1960, **14** (8), 1855–1856 (in English).—The composition of oxide films on heated copper wire has been studied by coulometry. The choice of electrolyte is important; 0·1M-KCl does not give a separate indication for Cu_2O and CuO; 0·1M-NH_4Cl does, but also partially dissolves the film. It is shown that 0·1M-KOH is satisfactory. *Procedure*—The electrodes consist of the wire under test and a silver - silver chloride reference electrode. Remove O from the electrolyte by passing a stream of N purified with pyrogallol and Cr^{II} salt soln. Pass a current of 670 μA per sq. cm, and record the potential - time curve. Calculate the quantities of Cu_2O and CuO from the breaks in the curve at $\simeq$0·85 and 1·1 V, respectively. G. Russell

1841. **Spectrophotometry of micro amounts of copper in samples in the alkali industry.** Hiroshi Awaya (Res. Lab., Tokuyama Soda Co., Tokuyama, Yamaguchi-ken). *Japan Analyst*, 1959, **8** (11), 752–753.—For the colorimetric determination of Cu in industrial hydrochloric acid, water glass or soda ash with diethyldithiocarbamate, the sample is neutralised and then oxidised with HNO_3; Fe, Ni and Mn are masked with EDTA. K. Saito

1842. **Spectrographic determination of high concentrations of copper in cupro-nickel and copper - manganese alloys.** N. K. Rudnevskiĭ, G. I. Golitsin and Yu. S. Kalinin. *Trudy Khim. i Khim. Tekhnol.*, 1958, (3), 584–586; *Ref. Zhur., Khim.*, 1960, (17), Abstr. No. 69,141.—A method is proposed for the determination of Cu in copper - nickel and copper - manganese alloys with a Cu content of 60 to 100%. The determination is carried out with two line pairs: Cu 2492·1 and 3093·9 A, and Cu 2400·1 and 3093·9 A. Spectra are excited in an a.c. arc. The error ranges from $\pm$1·0 to $\pm$2·2%, according to the alloy and the line pairs used. The greatest accuracy is attained with the lines Cu II 2400·1 and Cu I 3093·9 A. K. R. Cook

1843. **Determination of copper and lead in electrolytic tin and high-purity selenium by square-wave polarography.** Kamihiko Itsuki and Teru Kaji (Osaka Refinery, Mitsubishi Metal & Mining Co., Kita-ku). *Japan Analyst*, 1959, **8** (11), 703–707.—The square-wave polarography of Cu and Pb in $HClO_4$, preceded by the removal of Se and Sn with H_2SO_4 and HNO_3 and with HBr and Br, respectively (Barker and Jenkins, *Analyst*, 1952, **77**, 685; Ferrett and Milner, *Ibid.*, 1955, **80**, 132; 1956, **81**, 506) was examined. The wave heig increases with increase in superimposed potenti (15 to 30 mV) and the working curves are linear f $>$40 μg of Cu and $>$35 μg of Pb per 100 n *Procedure*—Heat the tin (1 g) first with HI (15 ml) and Br (1 ml) and then with HBr (10 n and HNO_3 (1:1) (10 ml). Dissolve the residue $HClO_4$ (60%) (5 ml) and water (20 ml), dilute 100 ml and record the polarogram between -0 and $-$0·4 V (for Cu) and $-$0·5 and $-$0·8 V (f Pb). With selenium, dissolve the sample (5 g) HNO_3 (40 ml), evaporate to dryness with H_2S (1:1) (10 ml) and treat the residue as described abov K. Saito

1844. **Use of mercury as collector for the separ tion of trace amounts of silver.** Shizo Hirano a Atsushi Mizuike (Inst. Tech.-anal. Chem., Fac. Engng, Tokyo Univ., Hongo). *Japan Analyst*, 195 **8** (11), 746–749.—When mercury (2 ml) is stirr in an ammoniacal soln. containing $<$10 μg of A and $\simeq$2 g of Cu and Fe, the Ag collects on t mercury within 30 min., while the Cu and remain in soln. The recovery of Ag was studied wi ^{110m}Ag and found to be $>$96%. This method also useful for an acid soln. containing M-H_2SO but the deposition of Ag is slower. K. Saito

1845. ***NN'*-Di(allylthiocarbamoyl)hydrazine (Da zin) as an analytical reagent. IV. Determination silver and its separation from other metals.** N. Dutt and K. P. Sen Sarma (Dept. of Chem., Uni Coll. of Sci., Calcutta, India). *Z. anal. Chem.*, 196 **177** (1), 7–9 (in English).—Silver is quant. pptd. silver "dalzinate," $Ag(C_8H_{13}N_4S_2)$, at pH 4·7 to 5 by the addition of an ethanolic soln. of the reage Zinc and Ni do not interfere, and may be determin in the filtrate; the separation of Ag from Cd and is effected in the presence of EDTA (disodium sal J. H. Waton

1846. **Determination of bismuth and lead crude silver by alternating-current polarograph** Kamihiko Itsuki and Mitsuyoshi Nagao (Mitsubis Metal & Mining Co., Shinkawasaki-cho, Kita-k Osaka). *Japan Analyst*, 1959, **8** (12), 800–804. Since Fe affects the a.c. polarography of Bi a Pb only slightly, these metals are co-pptd. wi $Fe(OH)_3$, dissolved in N-HCl and determin polarographically. When the superimposed pote tial is 20 mV, the peak heights at $-$0·15 a $-$0·48 V *vs.* the S.C.E. are respectively proportion to the concn. of Bi and Pb; As^{III}, Sb^{III} and Te interfere, but are removed by the pptn. proce *Procedure*—Dissolve the sample (2 g) in HNO_3 (1: (30 ml), dilute to 150 ml, add $(NH_4)_2SO_4.Fe_2(SO$ (10%) (10 ml), aq. NH_3 and ammonium carbona soln. (20%) (5 ml), boil, and filter. Wash the pp with hot water, then dissolve it in HCl (1:3 evaporate to a syrup, add HCl (1:1) (17 ml) a make up to 100 ml for polarography. K. Saito

1847. **Determination of bismuth and lead electrolytic silver by square-wave polarograph** Kamihiko Itsuki, Bunjiro Kobayashi and Kazu Nishino (Mitsubishi Metal & Mining Co., Shinkaw saki-cho, Kita-ku, Osaka). *Japan Analyst*, 195 **8** (12), 804–807.—The sensitivity of the meth previously described (Itsuki and Nagao, *Anal Abst* 1961, **8**, 1846) was increased by the use of squar wave polarography in N-HCl, so that 0·0001 0·001% of Bi and Pb in silver can be determine There is no interference from As^{III}, Sb^{III}, Te^{IV} or

l amount of Ag. About 20 mg of Fe^{III} suffices complete pptn. of Bi and Pb. The Fe^{III} is ced to Fe^{II} which does not interfere. *Procedure* ssolve the sample (5 g) in HNO_3 (1:1) (30 ml), Fe^{3+} (20 mg), aq. NH_3 and ammonium carbonate and filter. Dissolve the ppt. in HCl (1:1) nl), add hydrazine hydrochloride (1 g) and make 50 ml for polarography. K. Saito

48. **Gravimetric determination of gold(III) by rcaptoacetic (thioglycollic) acid.** Anil K. herji (Coates Chem. Lab., Louisiana State ., Baton Rouge, U.S.A.). *Anal. Chim. Acta*, , **23** (4), 325–327 (in English).—Gold may be t. pptd. from 6N-HCl by thioglycollic acid. he acid sample soln. (50 ml) is added thioglyc acid soln. (10%) (10 ml) and the mixture is ed on a steam bath for 30 min., and then set e for a further 30 min. The ppt. is collected in ntered glass crucible, washed six times with r, dried at 110° to 120° and weighed as $_2O_2SAu$. Results on 6 to 75 mg of Au are in $\pm 0{\cdot}8\%$ of the theoretical values. Platinum Zr are pptd. with the Au, but this can be ded by a preliminary extraction of the Au from loride soln. with isopropyl ether, and a backaction into 6N-HCl. A list is given of 20 ions do not interfere at concn. up to 4 times that of Au. Sulphate interferes, but small amounts of - are without effect. T. R. Andrew

849. **Determination of gold in electrolytic copper neutron activation analysis.** Shizo Hirano, ishi Mizuike and Koji Yamada (Inst. of Tech.-. Chem., Fac. of Engng, Tokyo Univ., Hongo, yo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, , **62** (10), 1494–1496.—Gold ($\simeq 0{\cdot}01$ p.p.m.) electrolytic copper ($\simeq 1$ g) is adsorbed on an n-exchange resin (Amberlite IRA-400) and irradiated in a reactor. The resin is ashed, the due is dissolved in aqua regia (0·3 ml) containing of Au as carrier and the ^{198}Au is extracted with l acetate (10 ml) for counting. The over-all very is $\simeq 95\%$ and the error is $<10\%$. The er limit of determination is $\simeq 0{\cdot}01$ μg when the tron flux is $\simeq 5 \times 10^{11}$ per sq. cm per sec. K. Saito

850. **Separation and determination of trace unts of gold in copper with mercury as collector.** o Hirano and Atsushi Mizuike (Inst. Tech.-l. Chem., Fac. of Engng, Tokyo Univ., Hongo, yo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, , **62** (10), 1497–1499.—When an ammoniacal . of copper is stirred with mercury, Au (a few is collected in the mercury, Cu remaining in the . For collecting 99% of the Au present in 50 ml soln., stirring for 30 min. is sufficient but, for ml of soln., stirring for 2 hr. is necessary. In absence of Cu, the amalgam formation is greatly rded. The mercury is evaporated off in a ent of N, and Au is determined photometrically *p*-dimethylaminobenzylidenerhodanine (**I**) in nol; the error is $\simeq 5\%$ for 1 to 100 p.p.m., $\simeq 10\%$ for 0·5 p.p.m., respectively, of Au. *cedure for crude copper*—Dissolve the sample to 200 mg) in HNO_3 (4 ml), heat with HCl (3 ml) 10 min., cool, add aq. NH_3 soln. (20 ml), stir mercury (2 ml) for 30 min.; remove the mer- and evaporate it in a current of N. Dissolve residue in aqua regia, dilute, filter, evaporate to ness, and again evaporate to dryness with aqua a (5 drops); dissolve the residue in HCl (1:100) ml) and NaCl soln. (4%) (5 ml), add NaF soln. (1%) (0·5 ml) and **I** in ethanol (0·0003%) (0·5 ml), make up to 10 ml and measure the extinction at 562 mμ after 15 min. K. Saito

1851. **Application of radioactivation analysis to gold ores of the Witwatersrand system.** N. H. Bailey and V. C. O. Schuler. *S. Afr. Ind. Chem.*, 1960, **14** (4), 67–71.—*Procedure*—A series of standards is prepared by mixing known weights of Au with SiO_2 powder. The standards (200 mg) and ore samples (200 mg) are weighed into polythene capsules and irradiated in a neutron pile. The γ-count of ^{198}Au is determined and from this the Au in the ore is calculated. Results compare satisfactorily with those by the normal fire assay. C. A. Slater

1852. **Gravimetric determination of small amounts of beryllium in ores and their products.** L. M. Moiseeva, N. M. Kuznetsova and I. I. Pal'shina. *Zhur. Anal. Khim.*, 1960, **15** (5), 561–563.—The method is based on the pptn. of Be with 2,2-dimethylhexane-3,5-dione (**I**). *Procedure*—Fuse 0·25 to 1 g of sample with 5 times its weight of KHF_2 in a platinum dish at 800° to 900°. Add 15 ml of H_2SO_4 (1:1) and evaporate. Transfer the residue to a beaker, add 10 ml of HCl (1:1) and dilute to 100 ml. Boil until dissolution is complete, then cool (Sn, if present, must be removed with H_2S). Add 40 ml of EDTA soln. (5%) to prevent interference by Fe, Al, UO_2^{2+}, Ti, Ce, Nd and Ca, adjust the pH to 7 to 8 with aq. NH_3, and add 20 to 30 ml of **I** (satd. aq. soln.) per mg of Be. Stir the mixture while adding 15 to 20 ml of H_2O and set it aside for 2 to 3 hr. Collect the ppt. in a sintered crucible (porosity 4), wash it with cold H_2O and dry at 45° to 55°. Phosphate, F^- and CO_3^{2-} do not interfere. J. W. Price

1853. **Determination of free carbon in beryllium metal (by ignition, with a gasometric finish).** U.K.A.E.A., Chemical Services Dept. (Springfields, Lancs.). Analytical Method IGO-AM/S-150, 1960. 10 pp.—After dissolution of the sample in H_2SO_4, the free carbon is filtered off and ignited in a stream of O. The CO_2 produced is collected and measured gasometrically. (*Cf.* U.K.A.E.A. Analytical Method IGO-AM/S-151 for details of apparatus and calculations.) G. P. Mitchell

1854. **Determination of combined carbon in beryllium metal (by ignition, with gasometric finish).** U.K.A.E.A., Chemical Services Dept. (Springfields, Lancs.). Analytical Method IGO-AM/S-151, 1960. 10 pp.—The method described, with details of apparatus, consists in dissolution of the sample in H_2SO_4, converting the methane and hydrogen produced into CO_2 and H_2O by passage over CuO at 850°, freezing out the CO_2, and finally determining the CO_2 gasometrically. (*Cf.* Bergholz, *Brit. Abstr. C*, 1953, 2.) G. P. Mitchell

1855. **Absorptiometric determination of silicon in beryllium.** U.K.A.E.A., Chemical Services Dept. (Springfields, Lancs.). Analytical Method IGO-AM/S-131, 1960. 5 pp.—After dissolution of the beryllium metal in Si-free NaOH soln., the Si is determined absorptiometrically by the molybdenum-blue method. G. P. Mitchell

1856. **Determination of oxygen in Magnox A12.** R. Berry, J. A. J. Walker and R. E. Johnson (U.K.A.E.A., Devel. and Engng Group, Res. and Devel. Branch, Culcheth). U.K.A.E.A. Report

DEG 111 (C), 1960. 15 pp.—Experiments are described in which the Mg was vacuum-sublimed at 800° from the sample (0·5 to 1·0 g); the O in the residue was determined after conversion into CO by heating in an iron bath at 1700° in a micro-vacuum fusion apparatus. Details are given of a procedure that is suitable for the analysis of Magnox A12 (a magnesium alloy) with a content of O from 10 to 200 p.p.m.; the standard deviation is ±10 p.p.m. G. J. HUNTER

1857. **Titration with EDTA (disodium salt) of magnesium and calcium in the presence of lead, zinc and mercury.** L. A. Vol'f. U.S.S.R. Pat. 128,191 (28th April, 1960).—Unithiol (Na 2,3-dimercapto-propanesulphonate) (**I**) is used as masking agent. For soln. containing 5 ml of 0·01N-Ca^{2+} and/or Mg^{2+} in the presence of Pb, Zn and Hg, use 10 to 20 ml of 0·25% **I** soln., 5 ml of buffer soln. (350 ml of 25% aq. NH_3 and 54 g of NH_4Cl per litre) and 10 drops of 0·4% ethanolic Eriochrome black T indicator, dilute to 100 ml and titrate with 0·01N-EDTA (disodium salt). C. D. KOPKIN

1858. **Application of selective adsorbent resins to analytical chemistry. II. Determination of calcium and magnesium in caustic soda.** Toshiharu Takagi and Hiroshi Imoto (Toyo Soda Mfg. Co., Tonda, Nanyo-machi, Tsuno-gun, Yamaguchi-ken). *Japan Analyst*, 1959, **8** (12), 782–785.—Calcium (<0·01%) and Mg (<0·01%) are quant. retained by a selective adsorbent resin (*Anal. Abstr.*, 1959, **6**, 3413) at pH 6 to 12, eluted with dil. HCl and titrated with EDTA. Iron and Al behave similarly, but are pptd. before ion exchange. *Procedure*—Dissolve the sample (50 g, or an equivalent amount of soln.) in water (200 ml), neutralise with HCl, render the soln. ammoniacal, filter and stir the filtrate with the resin (10 g) for 30 min. Collect and wash the resin; treat it with 0·5N-HCl (50 ml) for 20 min. and make the filtrate up to 250 ml. Titrate Ca and Mg in 100-ml aliquots with 0·005M-EDTA (disodium salt), with 3-hydroxy-4-(2-hydroxy-4-sulpho-1-naphthyl-azo)-2-naphthoic acid as indicator. K. SAITO

1859. **Use of 8-hydroxyquinoline and EDTA (disodium salt) for the rapid determination of calcium and magnesium in open-hearth slags of high phosphorus content.** N. N. Lapin, A. T. Slyusarev and N. S. Prilutskaya. *Sb. Nauch. Trud. Zhdanovsk. Metallurg. Inst.*, 1960, (5), 393–397; *Ref. Zhur., Khim.*, 1960, (18), Abstr. No. 73,051.—A complexometric method is developed for determining Ca and Mg after removal of Fe, Al, Mn and V by pptn. with 8-hydroxyquinoline in acetate buffer soln. (pH 6·2) without preliminary removal of silicic acid. C. D. KOPKIN

1860. **Stoicheiometry of titration of calcium, magnesium and manganese at low concentration with EDTA, with the metal indicators murexide and Eriochrome black T.** O. Gjems (Inst. of Soil Sci., Agric. Coll. of Norway, Vollebekk, Norway). *Analyst*, 1960, **85**, 738–744.—Titrations of Ca, Mg and Mn to determined colours of the indicators murexide and Eriochrome black T are compared with theoretically calculated values. Since the colours of murexide and Eriochrome black T are not very stable at high pH, artificial reference colour standards were prepared with gentian violet and methyl red for the titration of Ca with murexide, and with methylene blue and methyl red for the titration of Mg and Mn with Eriochrome black T. The titration of Ca is stoicheiometric within the accuracy of the titration. In the titration of there is a difference between the observed calculated figures, probably due to low reaction since the difference is reduced with increase of te Titration values for Mn agree well with theoret figures, but a small difference between results room and higher temp. suggests a low reaction r In the presence of Ca and Mg, Mn can be calcula from the difference between the titre for Ca, Mg Mn together, with Eriochrome black T indica and the titre for Ca plus Mg after Mn has b removed with Na diethyldithiocarbamate and C A. O. JONE

1861. **Indicator for the determination of calci** I. M. Korenman, V. G. Ganina and E. I. Lei *Trudy Khim. i Khim. Tekhnol.*, 1959, (1), 108–1 *Ref. Zhur., Khim.*, 1960, (17), Abstr. No. 69,146 Acid Monochrome Bordeaux S forms coloured co plexes with the cations of alkaline-earth metals pH 10, *viz*, violet with Ba, pink with Sr, yel with Ca and orange with Mg: the control is ra berry red; Ca gives the most sensitive and select reaction (limiting concn. 1 in 2×10^5). The col change at the end-point with EDTA is far shar than with murexide. K. R. Coo

1862. **Polarographic determination of calcium solutions and plant tissue.** A. E. Petrov-Spiridon *Izv. Timiryazev. Selsko. Akad.*, 1960, (1), 236–2 *Ref. Zhur., Khim.*, 1960, (17), Abstr. No. 69,148 The method is based on the decrease in the hei of the picrolonic acid (**I**) wave due to the pptn. insol. Ca picrolonate. The decrease is proportio to the Ca concn. in the range 0 to 25 mg per Add **I** soln. (1 g of **I** per 500 to 600 ml of H (0·5 ml) to the test soln. (2 ml), set aside for ≃12 and filter. Add acetate buffer soln. (pH 3·6) (3 to the filtrate and polarograph it at 0 to −0·8 *vs.* the S.C.E. To determine Ca in plant tiss moisten the sample (10 to 50 mg) with 0·2N-H (a few drops), ash it at 500° to 600° for 3 to 4 h add 0·2N-HCl (2 to 3 drops), neutralise with 0·1 KOH or NaOH to methyl red, dilute to 10 ml a proceed as described above. With a Ca concn. 10 to 25 mg per ml the error is <5%. K. R. Coo

1863. **Inspection of calcium fluoride.** U.K.A.E. Production Group (Windscale, Cumberland, En land). U.K.A.E.A. Report PG 114 (W), 19 18 pp.—Full details are given for the preliminar separation, when necessary, and the determinati of (*i*) Ca, gravimetrically as $CaCO_3$; (*ii*) CO_3^{2-}, weighing of CO_2; (*iii*) Cl^-, by nephelometry AgCl; (*iv*) Cr, by absorptiometry of its complex w diphenylcarbazide; (*v*) Fe, by absorptiometry of complex with thioglycollic acid; (*vi*) Si, by absorpt metry after conversion into molybdenum blu and (*vii*) SO_4^{2-}, gravimetrically as $BaSO_4$. The l of weight on ignition is also determined, and t material is graded by sieving. G. J. HUNTER

1864. **Separation of strontium and calcium w the use of EDTA. Determination of strontium apatites.** L. I. Afanas'ieva. *Zhur. Anal. Khi* 1960, **15** (5), 564–567.—Strontium (1 to 20 mg) separated from up to 200 mg of Ca by pptn. $SrSO_4$ in the presence of EDTA (**I**). *Procedure* Dissolve up to 150 mg of the mixed oxides in t minimum amount of HNO_3 or HCl. Add 800 m of **I** and neutralise to methyl red with aq. NH Add 10 ml of $(NH_4)_2SO_4$ soln. (10%) and dilute 30 to 35 ml. Heat and add dil. H_2SO_4 to t

pearance of a permanent rose colour (pH ≃4·5). For large amounts of Sr (Sr:Ca > 1:5), set the n. aside for 30 min. at 70° to 80°. Filter after r. and wash the ppt. with H_2O containing a little $H_4)_2SO_4$. Determine by igniting and weighing $SrSO_4$, or by dissolving in excess of ammoniacal and titrating with $MgCl_2$ soln. (*ii*) For small ounts of Sr (Sr ≮1 mg; Ca ≯200 mg), add 10 ml acetate buffer (pH 4·5) and 50 ml of ethanol %). Set aside for 24 hr., filter, and wash the t. with ethanol (50%) containing $(NH_4)_2SO_4$. nite and weigh as $SrSO_4$. Calcium can be determined in either filtrate by pptn. as oxalate, or culated by difference. Procedure (*ii*) can be ed for the determination of Sr in apatites, with ·2-g sample. J. W. Price

1865. **Chemico-spectrographic determination of purities in strontium and barium.** M. A. Notkina d B. M. Dobkina (State Sci. Res. and Development Inst. of the Rare Metals Industry). *Zavod. b.*, 1960, **26** (10), 1126–1128.—The sample is ssolved in CO_2-free water, the soln. is neutralised th HCl and the metallic impurities Bi, Fe, Cd, , Cu, Pb, Sb and Zn are extracted by means of diethyldithiocarbamate soln. and $CHCl_3$. The tract is evaporated in the presence of carbon wder, and the residue is subjected to spectroaphic analysis. G. S. Smith

1866. **Colour reactions for barium and calcium.** M. Korenman and V. G. Ganina. *Trudy Khim. i him. Tekhnol.*, 1958, (3), 545–551; *Ref. Zhur., him.*, 1960, (17), Abstr. No. 69,123.—Colour actions of Ba^{2+} with Lacquer Scarlet S and of $^{2+}$ with 3-(*o*-hydroxyphenylazo)chromotropic acid, (4-sulphophenylazo)chromotropic acid and Acid onochrome Bordeaux S are described.
K. R. Cook

1867. **Zinc colour reactions.** F. S. Frum, G. G. rasnova and G. V. Nazarova. *Trudy Khim. i him. Tekhnol.*, 1958, (3), 579–580; *Ref. Zhur., him.*, 1960, (17), Abstr. No. 69,152.—In the esence of $K_4Fe(CN)_6$, Zn gives sensitive colour actions with methylene blue (**I**) and with brilliant een (**II**); with **I**, 1 μg of Zn per ml can be detected; , Pb and Ag do not interfere; Cu, Fe, Co, Cd and n should first be separated by alkaline pptn. he reaction can be used for the colorimetric deterination of Zn by the use of standards; the mean ror is ≃2%. With **II**, 8 μg of Zn per ml can be tected. K. R. Cook

1868. **Determination of zinc in indium and nickel.** G. Towndrow, R. Hutchinson and H. W. Webb ohnson, Matthey & Co. Ltd., Res. Lab., Exhibion Grounds, Wembley, England). *Analyst*, 1960, , 769–770.—An aliquot of a diluted soln. of the mple in dil. HCl is evaporated with $FeCl_3$ soln. the residue, dissolved in the minimum amount of Cl, is added 0·5 ml of n-butanol and the mixture is plied to a cellulose column, which is eluted with butanol - HCl until the Fe band has been completely eluted, when all the Zn will be in the eluate. he residue from evaporation of the eluate is aporated again with $HClO_4$ and then dissolved in l. HCl; after addition of aq. NH_3 and specified ounts of a base electrolyte containing aq. NH_3, a_2SO_3 and gelatin, the soln. is centrifuged and the is determined polarographically in the supertant liquid. To determine Zn in nickel, the sample dissolved in dil. HCl, $CoCl_2$ soln. and $FeCl_3$ soln. are lded and the procedure is then as described above. The blue Co band follows the Fe band down the column and appears to act as a barrier to the Ni, which has a tendency to trail. A. O. Jones

1869. **Use of a stationary mercury electrode for micro-analytical determinations. [Determination of cadmium.]** Yu. M. Kargin and E. A. Berdnikov (Kazan State Univ.). *Zavod. Lab.*, 1960, **26** (9), 1078–1079.—The electrode consists of a platinum needle (fused into a glass tube) which is treated first with conc. HNO_3, then with a soln. of $FeSO_4$ and H_2SO_4, and washed with water. The electrode is immersed in $Hg(NO_3)_2$ soln., prepared by mixing the saturated soln. with eight times its vol. of water, and Hg is deposited on it electrolytically. It is then washed with hot and cold water. Two such electrodes are used in a scheme of differential oscillographic polarography. The soln. to be analysed, *e.g.*, a soln. containing Cd, is electrolysed with an applied e.m.f. of ≃ 5 V. The amount deposited is then determined from the oxidation current of the amalgam obtained. The method can be used for the determination of concn. of ≃0·001 μg per ml. G. S. Smith

1870. **Alternating-current polarisation titration with two hanging-mercury-drop electrodes—application to chelatometric titration of cadmium.** Toyokichi Kitagawa (Dept. of Chem., Fac. of Educ., Kobe Univ.). *Bull. Chem. Soc. Japan*, 1960, **33** (8), 1124–1127 (in English).—A mixture of cadmium nitrate soln. (≃50 ml), M-ammonium acetate - acetic acid buffer soln. (pH 4·3 to 5·0) (50 ml), 0·1% gelatin soln. (1 ml) and 0·05M-Hg^{II} - EDTA (10 ml), was titrated with 10^{-1} or 10^{-2}M-EDTA (disodium salt). The frequency of the square or sine wave was 25 c/s from a Kikusui Type CR oscillator, and the a.c. was 20 to 25 μA. It is concluded that the end-point is indicated by the change of the mercuric ion concn. near the equivalence point. The error for Cd concn. of 10^{-2} to 10^{-3}M was within 0·9%.
E. M. Mayes

1871. **1,2-Dimorpholinoethane as a reagent in quantitative analysis. I. Gravimetric determination of mercury.** E. Asmus and K. Ohls (Lehrgebiet Anal. Chem., Tech. Univ., Berlin). *Z. anal. Chem.*, 1960, **177** (2), 100–109 (in German).—1,2-Dimorpholinoethane (**I**) gives with Hg^{II}, in the presence of Br^- or I^-, insol. compounds, namely, **I**.HgI_2.2HI at pH ≯3, and **I**.HgI_2 and **I**.$HgBr_2$ at pH 6 to 7. These products may be dried at 110° and weighed. Recoveries of 0·1 to 1 g of Hg are within 0·1%. The pptn. is quant. under any conditions in the presence of 10 g of Li, Na, K, Mg, Ca, Sr, Ba, Al, Cr, Mn, Co or Ni, or 1 g of As^{III}, Zn, Mo or W; 1 g of Fe^{III}, Sb, Ti or Cd may be present if pptn. is made from I^- soln. at pH 3, while 10 g of Cu or 1 g of Cd may be tolerated when pptn. is made from Br^- soln. at pH 6 to 7.
T. R. Andrew

1872. ***NN'*-Di(allylthiocarbamoyl)hydrazine (Dalzin) as an analytical reagent. III. Determination of mercury and its separation from other metals.** N. K. Dutt and K. P. Sen Sarma (Dept. of Chem., Univ. Coll. of Sci., Calcutta, India). *Z. anal. Chem.*, 1960, **177** (1), 4–7 (in English).—Mercury is quant. pptd. as mercury "dalzinate," $Hg(C_8H_{12}N_4S_2)$, at pH 3·0 to 3·5 by the addition of an aq. soln. of the sodium salt of the reagent. Zinc, Ni and Pb do not interfere, and may be determined in the filtrate; the separation of Hg from Cd, Bi and Cu is effected in the presence of EDTA (disodium salt).
J. H. Waton

1873. **The mass-spectrometric analysis of the isotopic composition of elementary boron.** Yu. A. Zonov (State Inst. of Appl. Chem., Leningrad). *Zhur. Anal. Khim.*, 1960, **15** (5), 643–645.—In the process of evaporation of B from a ribbon evaporator the normal fractionation of isotopes occurs, and this has little or no effect on the accuracy of determination of the isotopic composition of the sample.
J. W. Price

1874. **General chemical - spectrographic method for the determination of boron in iron and nickel-base alloys.** R. F. Anderson (Res. Lab., The International Nickel Co. Inc., Bayonne, N.J., U.S.A.). *Appl. Spectroscopy*, 1960, **14** (5), 123–125.—Base metals are removed from the sample by mercury cathode separation, when boron remains in the electrolyte. A uniform matrix for all types of alloy is provided by the addition of a standard copper soln. The resulting soln. is evaporated to dryness and baked to oxide, which is mixed (1 : 1) with pure graphite and arced in a crater electrode at 10 amp. d.c. One set of synthetic standards covers all types of alloy. Good agreement is shown between chemical and spectrographic results and the coeff. of variation is $\simeq 5$ to 10% over the range 0·001 to 0·02% of B.
P. T. Beale

1875. **Anthraquinone derivatives as reagents for boric acid.** I. M. Korenman and N. V. Kurina. *Trudy Khim. i Khim. Tekhnol.*, 1958, (3), 573–577; *Ref. Zhur., Khim.*, 1960, (17), Abstr. No. 69,194.—Of the 13 derivatives studied the most sensitive and characteristic reactions with H_3BO_3 were given by Alizarin blue (C.I. Mordant Blue 27), Alizarin Sapphirol SE (C.I. Acid Blue 43), Alizarin Sapphirol A (C.I. Acid Blue 25), Indanthrene Brilliant violet BBK, Celliton Fast blue-green B and Acid blue-black anthraquinone S; F^-, VO_3^- and WO_4^{2-} do not interfere. The reaction sensitivities and specificities are determined.
K. R. Cook

1876. **Chromoxan pure blue B as a reagent for aluminium.** F. P. Sheyanova and V. P. Malenskaya. *Trudy Khim. i Khim. Tekhnol.*, 1958, (3), 552–556; *Ref. Zhur., Khim.*, 1960, (17), Abstr. No. 69,157.—It is shown that Chromoxan pure blue B (C.I. Mordant Blue 1) (**I**) gives a violet colour with Al^{3+} at pH 4 to 6. The detectable minimum is 5 μg per ml; Zn, Mg, Ni, Cd, Pb, Cr and Mn do not interfere; Fe^{3+} or Cu^{2+} also form coloured compounds with **I**, and in their presence a reducing agent should be added to the test soln. (ascorbic acid to reduce Fe^{3+} and NH_4SCN to reduce Cu^{2+}). The reaction is suitable for the detection of Al in certain alloys.
K. R. Cook

1877. **Complexometric determination of aluminium [in magnesium alloys] with amperometric indication of the end-point.** V. M. Babenyshev and O. M. Kuznetsova. *Zhur. Anal. Khim.*, 1960, **15** (5), 568–572.—*Procedure*—Dissolve 1 g of sample in 20 ml of HCl (1 : 1) and dilute to 200 ml. To a 1-ml aliquot add 2 ml of 0·1M-EDTA and 0·2 ml of ammonium acetate soln. (5%) and dilute to 25 ml. Titrate amperometrically with 0·1M-$FeCl_3$, with a rotating platinum anode (700 r.p.m.), without applied e.m.f., and a S.C.E. connected to the titration beaker through an agar bridge. Greater speed and accuracy in detecting the end-point can be obtained by measuring the time to the first movement of the galvanometer needle, and by using a special burette to ensure a constant rate of addition of the $FeCl_3$ soln.
J. W. Price

1878. **Separation and determination of tr amounts of heavy metals in high-purity aluminiu** Shizo Hirano, Atsushi Mizuike and Yoshio Ii (Inst. Tech.-anal. Chem., Fac. of Engng, Tok Univ., Hongo, Tokyo). *J. Chem. Soc. Jap Ind. Chem. Sect.*, 1959, **62** (10), 1491–1494.—T use of Kraus's ion-exchange method and of mercu cathode electrolysis was examined. The R_F valu of Co, Fe and Zn appear to be considerably increas in the presence of a large amount of Al; Co and are eluted with 4N-HCl and N-HNO_3 and determin photometrically with nitroso-R salt and th glycollic acid, respectively; Cu, Cd and Zn a eluted with N-HNO_3 and determined pola graphically. *Procedure for ion exchange*—Dissol the sample (0·4 g) in 9N-HCl (50 ml) and wa (30 ml) in the presence of a trace of $NiCl_2$ (50 μg) increase the rate of dissolution. Add 9N-HCl ml), boil with H_2O_2 (30%) (a few drops), co dilute to $\simeq 75$ ml and pass through a colun (diameter 2 cm, length 5 cm) of Amberlite IRA-4 (60 to 100 mesh) at a rate of 1 ml per min.; wa with 9N-HCl (3 ml) and elute Co with 4N-HCl (40 m and Fe with N-HNO_3 (40 ml), and then Cu, Cd a Zn with N-HNO_3 (45 ml). *Electrolysis*—A soln. the sample in dil. H_2SO_4 (1·5 ml) is electrolysed wi a mercury cathode (1 to 2 amp., 12 to 17 V) for 5 h the mercury is evaporated off in a current of N 350° and the residue is dissolved in aqua regia f the analysis.
K. Saito

1879. **Systematic neutron-irradiation analysis very-high-purity aluminium and iron.** J. Gaitt (Centre d'Ét. de Chim. Métall., C.N.R.S., Vitry-su Seine, France). *Ann. Chim., Paris*, 1960, **5** (9–1 1219–1271.—For the determination of impuriti in commercial aluminium and iron (prepared electrolysis and refined by zone-melting), t procedure of Albert (*Ibid.*, 1956, **1**, 827) has be improved to achieve chemical separation (by grou and sub-groups) of the radio-isotopes formed neutron irradiation (2×10^{12} neutrons per sq. c per sec. for 145 hr.) and subsequent measureme of their activity, by decay-period or γ-spectrometr in comparison with known standards. The radi isotopes of the rare-earth elements are separat by elution from Dowex-50 resin with ammoniu lactate soln. at 90°. About 40 elements can determined in concn. of parts per 10^6 or 10^8, in 1-g sample, whilst a further 12 can be determin in separate samples, *e.g.*, Ba, S, P and Cl (0·01 0·05 p.p.m.) in zone-melt refined metals. T limit of sensitivity is 0·0001 p.p.m., and it is possib to determine 0·0004 p.p.m. of As and 0·002 0·003 p.p.m. of Sb, Th or U in the 60 p.p.m. impurities in electrolytic aluminium. The purit grading of refined aluminium and iron, as deduc from the chemical analyses, is in complete agreeme with that based on measurements of residual res tance at low-temp.
W. J. Baker

1880. **Composite procedure for the analysis aluminium bronze alloys.** M. Freegarde and Allen (Bragg Lab., Naval Ordnance Inspecti Estab., Janson St., Sheffield, England). *Analy* 1960, **85**, 731–735.—The sample is heated first wi H_2SO_4 and HNO_3 and then to fuming with HC and the liquid is diluted, boiled and filtered. T $PbSO_4$ in the ppt. is dissolved in ammonium aceta soln., an ammoniacal aliquot of the soln. is extract with dithizone in $CHCl_3$ and the extinction of t extract is measured at 535 mμ. The residual pp of SiO_2 is ignited and weighed. Copper is dete mined electrolytically in the original filtrate a

in an aliquot of the Cu-free electrolyte by suring the extinction of the complex with EDTA 520 mμ. Nickel is determined spectrophotorically at 550 mμ in an aliquot of the Cu-free trolyte by means of the complex formed with xime. For Al, a buffered aliquot of the electrolyte reated with an excess of EDTA soln., the excess eutralised by titration with 0·1N-$CuSO_4$ to the et end-point of 1-(2-pyridylazo)-2-naphthol ., NaF soln. is added and the boiled and cooled id is titrated with 0·1N-$CuSO_4$ to the blue end-nt. Zinc is determined by measurement of the inction of the complex with dithizone at 535 mμ. nganese is determined by oxidation to MnO_4^- spectrophotometric measurement. Tin is ermined polarographically. A. O. Jones

881. **New volumetric method of analysis of minate solutions.** A. A. Bashkirtseva and L. D. idnikova (S. M. Kirov Ural Polytech. Inst.). *od. Lab.*, 1960, **26** (10), 1107–1109.—For the ermination of total alkali, alkali carbonate and in the one soln., the sample containing $>0·12$ g Al_2O_3 is treated with 0·1M-EDTA (disodium salt) an amount sufficient to combine with the Al and leave an excess of 1 to 3 ml. After dilution to ween 60 and 80 ml, the soln. is titrated with N-HCl in the presence of phenolphthalein. An ess of 0·5N-HCl is then added and the soln. is led to decompose the bicarbonate. The excess of l is titrated with 0·5N-NaOH. The cooled soln. reated with 5 ml of a buffer soln. (pH 5·5) and l of 10% Na sulphosalicylate soln. and titrated h 0·02M-$FeCl_3$ until a brick-yellow colour persists 1 to 2 min. This titration measures the excess EDTA. From the volume of EDTA soln. used l the results of the three titrations, the required ntents are calculated. G. S. Smith

882. **Spectrographic analysis of indium metal impurities.** H. M. Hyman (Semiconductor and terials Div., R.C.A., Somerville, N.J., U.S.A.). *pl. Spectroscopy*, 1960, **14** (5), 125–127.—Eight purities (Cu, Ga, Fe, Pb, Ni, Ag, Sn and Zn) are ermined within the range 0·0005 to 0·1% by ans of a solution method, in which a centre-post ctrode, on which the sample is dried, and a high-ltage a.c. arc are used. Synthetic standards are d and Bi is added as an internal standard for the ermination of Pb, Sn, Ag, Cu and Zn. Repeat ctra indicate an accuracy of $\simeq$15% of the ntent. P. T. Beale

883. **Spectrophotometric determination of traces nickel in pure indium and aluminium.** V. M. shkova, V. M. Bochkova and L. I. Lazareva . V. Lomonosov Moscow State Univ.). *Zhur. al. Khim.*, 1960, **15** (5), 610–613.—*Procedure with minium*—Dissolve 1 g of sample in HNO_3 in a artz vessel and evaporate to dryness. Dissolve e residue in 30 ml of Na tartrate soln. (30%) add l of furil α-dioxime soln. (0·1%) (**I**) and adjust pH 8·7 to 9·3. Extract with $CHCl_3$ (2 × 2·5 ml) d remove Cu from the combined extracts by aking with 10 ml of aq. NH_3 (1:40). Make up to l and measure the extinction at 438 mμ. *ocedure with indium*—Dissolve 1 g of sample as scribed above, evaporate, and dissolve the residue 20 ml of tartaric acid soln. (20%). Add 5 ml of **I** d 10 ml of aq. NH_3, adjust to pH 8·7 to 9·3 and ntinue as described above. As little as 5 parts r 10^8 of Ni can be determined with an accuracy thin ±25%. J. W. Price

1884. **Analytical chemistry of thallium. V. Complexometric indicators for tervalent thallium.** A. I. Busev and V. G. Tiptsova (M. V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1960, **15** (5), 573–580.—The composition of compounds formed by Tl^{III} with 1-(2-pyridylazo)-2-naphthol (**I**), 4-(2-pyridylazo)resorcinol and xylenol orange and their stability constants were determined and molecular extinction coeff. calculated. Optimum values of pH for the use of these indicators in the titration of Tl^{III} with EDTA were 1·8, 1·7 and 2·0, respectively; **I** was the most selective.

J. W. Price

1885. **Analytical chemistry of the rare-earth elements. Simple chromatographic procedure for the quantitative analysis of mixtures.** G. Brunisholz and J. P. Quinche (Lab. de Chim. Min. et Anal., Univ., Lausanne, Switzerland). *Chimia*, 1960, **14** (10), 343–352 (in German).—The chemistry and uses of the rare earths are discussed, together with previous analytical methods for their separation. The chromatographic separation is carried out on a sulphonic acid ion-exchange resin in the Cu^{2+} - H^+ form (Marsh, *J. Chem. Soc.*, 1957, 978), in a column of 2 mm diam. The eluting solvent is a soln. of EDTA (triammonium salt) buffered with ammonium formate or acetate. The elements appear, in the order of the stability of their complexes, as sharply defined zones, visible in u.v. light. If a tube of uniform bore is used, and the resin is packed uniformly, then the length of any zone will be a measure of the amount of rare-earth element giving rise to it; the length of each zone is measured, and expressed as a percentage of the total zone length. The elements that can be separated are (from the top downwards) La, Ce, Pr, Nd, Sm plus Eu, Gd plus Tb plus Y, Dy, Ho, Er, Tm, and Yb plus Lu; Sc does not appear, since its very stable EDTA complex is not decomposed by the Cu in the resin. Other metals may be separated by the procedure, and, if present, may interfere. Typical analyses are presented, and the preparation of reagents and apparatus is described. H. M.

1886. **New chelatometric methods for the [determination of the] rare earths.** G. Asensi Mora. *Inf. Quim. Anal.*, 1960, **14** (5), 121–136.—Bromopyrogallol red has been suggested as an indicator for the direct titration of rare-earth metals in acetate-buffered soln. The colour change from violet to red is not very clear and a new indicator, catechol green (3,6-dihydroxy-9-*o*-sulphophenylfluorene-4,5-quinone) is proposed. This forms olive-green complexes with rare-earth elements, which are decomposed by EDTA (disodium salt) to liberate the free indicator, which is blue. Recoveries of Y, La, Nd, Gd and Er were within 0·80, 0·95, 1·60, 1·28 and 1·20%, respectively, of the amount taken.

G. H. Foxley

1887. **Spectrophotometric determination of erbium and holmium in solutions.** I. I. Antipova-Karataeva and Yu. I. Kutsenko (V. I. Vernadsky Inst. of Geochem. and Anal. Chem., Acad. of Sci., USSR, Moscow). *Zhur. Anal. Khim.*, 1960, **15** (5), 581–586.—Erbium and Ho as chlorides in aq. soln. can be determined over the range 0·05 to 1·0% by measurement of their extinctions at 522·2 mμ and 536·8 mμ, respectively. Dysprosium and other rare-earth metals do not interfere. In ethanolic soln. the best wavelengths for measurement are 520·8 mμ (for Er) and 537·0 mμ (for Ho).

J. W. Price

1888. **Determination of actinium and radium by the copper-spark spectrographic method.** F. T. Birks (A.E.R.E., Harwell, England). *Spectrochim. Acta*, 1960, **16** (9), 1016–1021.—A copper-spark method has been developed for the determination of Ac and Ra in mixtures and in liquid wastes. Spectra are recorded in a Littrow spectrograph in the region 2800 to 5000 A, and sensitive lines are listed, together with interfering elements. Molybdenum is added as an internal standard by evaporating 2 μg of Mo on to each copper electrode before adding the sample soln. The limits of detection are 0·02 μg of Ac and 0·003 μg of Ra in approx. 0·05 ml of soln. A coeff. of variation of approx. 7% is obtained for the determination of Ac.

P. T. BEALE

1889. **Improved method for the determination of carbon dioxide.** Eiji Otsuka, Toru Takahashi and Shinji Yoshimura (Toyo Koatsu Ind., Totsuka-ku, Yokohama). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1960, **63** (2), 252–254.—The large error (5 to 15%) involved in the volumetric determination of CO_2 in a liquid sample is due to the dissolution of CO_2 in the soln. acidified with H_2SO_4. This effect is eliminated by passing a current of air (≃50 ml in 1 min.) through the sample in the reaction vessel, which is placed in a boiling-water bath. The sample (2 to 5 ml) in the reaction vessel is acidified with conc. H_2SO_4 and the stream of air is passed after 1 min. The error is <1% and the time required for a determination is 10 min. K. SAITO

1890. **Sodium carbonate, anhydrous (photographic grade).** British Standards Institution (2 Park Street, London, W.1). B.S. 3305:1960. 9 pp.—Tests for purity and a method of assay are given.

1891. **The determination of alkali carbonate in the presence of alkali hydroxide.** L. Szekeres and E. Bakács-Polgár (Chem. Inst., Hochschule f. Veterinärwiss., Budapest). *Z. anal. Chem.*, 1960, **177** (2), 89–91 (in German).—Mixtures of Na_2CO_3 and NaOH (in 10:1 to 3:10 molar ratios) may be assayed with a precision of ≃ 0·5% by titrating total alkali acidimetrically on one portion and adding a measured excess of $Sr(NO_3)_2$ soln. (20 ml of 0·1M) to a second portion (0·05 to 0·10 g of Na_2CO_3), filtering off the ppt. of $SrCO_3$, adding ethanol to the filtrate (to a final concn. of ≃30% v/v), and titrating the excess of Sr with EDTA (disodium salt), with methylthymol blue as indicator.

T. R. ANDREW

1892. **The determination of silica. III. Interference from aluminium, iron and fluorine in the spectrophotometric determination of silica.** L. H. Andersson (Inst. Chem., Univ., Uppsala, Sweden). *Acta Chem. Scand.*, 1960, **14** (7), 1571–1579 (in English).—*Procedure*—Extinctions are measured at 20° and 400 mμ. The silica soln. is mixed with the soln. of the interfering element in a polythene vessel, the pH is adjusted to the required value and sodium molybdate soln. of the same pH is added. The pH is again adjusted and the liquid is transferred to a polythene vessel on a steam bath. After heating for 6 hr. the vessel is placed in a thermostat at 20° and the soln. is diluted to a known vol. for extinction measurements. *Interference from NaF*—The presence of NaF increases the minimum concn. of molybdate required to obtain the full molybdosilicate colour. Decreasing the pH increases the effective molybdate concn. The extinctions of blanks vary greatly at low pH values. *Interference from Al*—The minimum amount of sodium mol date necessary for the full development of the col is higher than when no Al has been added. T useful pH range is restricted and is a function of molybdate concn. If the pH is >2·5, a ppt formed. In the pH range 0·5 to 1·5, Si may determined by the molybdosilicate method by add $AlCl_3$, which forms complexes with F^-. The should be somewhat less than 1·5. In the vicin of pH 1·5, the extinction is a function of the pH a of the Al concn. *Interference from Fe^{3+}*—The is adjusted with HCl to 1·5 and the liquid is hea on a steam bath for 3 hr. It is then transferred a thermostat at 20° and the extinction is measu as soon as the liquid is clear. The extinction of soln. in contact with the ppt. is a function of ti If only a small amount of Fe is present, the should be ≃ 2. The extinction is not influen by small changes in pH and Fe^{3+} concn. The of F^- for complexing Fe^{3+} is unsuccessful. *Ef of anions*—If SO_4^{2-}, NO_3^- or ClO_4^- are substitu for Cl^-, the results are similar, except that ppt. formed more easily in HNO_3 and $HClO_4$ soln. a the interference of small amounts of Fe is m pronounced in H_2SO_4 soln. *Extraction of Fe*— no F^- are present, the extraction is made at pH but, if F^- are present, at pH 7·5 in platinu Acetylacetone is used as complexing agent and C as solvent. When the Fe has been removed extraction of the aq. phase (≃ 35 ml) with C (3 × 5 ml), 0·5 g of Na_2CO_3 is added to the phase, the soln. is evaporated to dryness in platinu and the organic compounds are destroyed by fusi The melt is dissolved in H_2O and the Si determi spectrophotometrically. D. C. ARMSWORTH

1893. **Gravimetric method of determining lead [means of resorcinol].** P. Shtefznesku and Shtefznesku ("Petrokhim" Inst., Ploesti, Romani *Zavod. Lab.*, 1960, **26** (9), 1080.—The soln. (20 30 ml) containing Pb is treated with a reagent so prepared by dissolving 10 g of resorcinol in 10 of 20% aq. NH_3 and diluting to 100 ml. The so is added dropwise until pptn. is complete. Af addition of a further 2 to 3 ml and setting aside the ppt. to coagulate, the ppt. is collected in filter crucible and washed with water, then w ethanol, and finally with ethyl ether, dried a weighed. The ppt. has the composition $C_6H_4O_2$ Tin, Bi and Sb do not interfere. The reagent gi a green colour with Pb^{2+}, and is sensitive to presence of 0·03% of Pb. G. S. SMITH

1894. **Extraction - photometric determination traces of lead in reagents of high purity [thalli and sodium iodides].** A. M. Bulgakova and A. Volkova (All-Union Sci. Res. Inst. of Che Reagents, Kharkov Branch). *Zhur. Anal. Khi* 1960, **15** (5) 591–594.—*Procedure with thalli iodide*—Dissolve 5 g of sample in H_2O (5 ml), H_2S (1 ml) and HNO_3 (2 ml) and evaporate to fum Dissolve the residue in 150 ml of H_2O, add H (10 ml) and H_2O_2 (10 ml) and heat to remo iodine. Cool, and add more H_2O_2 to oxidise the Boil off the excess of H_2O_2 and evaporate to ≃3 Add 6N-HCl (20 ml) and extract (×3) with i pentyl ether. Neutralise the aq. layer, add 1 NH_4SCN soln. (5 ml) and pyridine (1 ml), a extract with $CHCl_3$ (10 ml) to remove Cu, Ni, Mn, Co and Fe. Wash the aq. layer with CH add citrate buffer (pH 8) (20 ml) and 5% diethyldithiocarbamate soln. (**I**) (10 ml) and extra Pb by shaking with $CHCl_3$ (10 ml). Wash

ss of **I** with H_2O, and replace the Pb in the plex with Cu by shaking with a 0·01% soln. of a er salt in citrate buffer (10 ml). Make up to l with $CHCl_3$ and measure the extinction of the **I** complex. The limit of determination is 10^{-5} % of Pb. *Procedure with sodium iodide*— plve 10 g of sample in H_2O (30 ml), add 1% $_2O_3$ soln. (3 ml) and neutralise with HCl, add SCN and pyridine and continue as described e. The limit of determination is 3×10^{-5} % J. W. Price

95. **Chemical and spectrographic method for the rmination of impurities in lead.** A. G. Karabash, . Bondarenko, G. G. Morozova and Sh. I. ılayev. *Zhur. Anal. Khim.*, 1960, **15** (5), 627.—In this method for the analysis of lead its compounds, a preliminary chemical con- ration of impurities is followed by spectro- hic analysis. For comparison, the original ple is examined spectrographically. *Pro- re*—Dissolve 10 g of sample in 6N-HNO_3 (45 ml) dilute to 100 ml. Add 35 ml of H_2SO_4 (10%), to 80° and set aside for 3 hr. at room temp. nt, heat the $PbSO_4$ with 6N-HNO_3 (40 ml) for Decant and combine the soln. Evaporate to ess and ignite at 550° for 30 min. Sodium, Mg, Al, Ti, V, Cr, Mn, Mo, Co, Ni, Sb, Zn, Cd, Cu, Bi, Sn, Ag, In, Te and As in concn. 10^{-1} to 10^{-6} % not co-pptd. with the $PbSO_4$. A 30 to 100-fold entration of impurities was obtained. The entrate was analysed spectrographically. The tivity was 10^{-4} to 10^{-6} % and the relative error ±20%. J. W. Price

96. **Determination of copper in lead and lead e-sheathing alloys.** J. H. Thompson and M. J. enscroft (Materials Section, Test and Inspection ch, Post Office Engng Dept., Fordrough Lane, ningham, England). *Analyst*, 1960, **85**, 735–738. ne sample (0·25 g) is dissolved in a mixture of c acid and H_2O_2, the soln. is boiled gently almost yness, 6N-HCl is added and the liquid is warmed issolve $PbCl_2$ and finally boiled. The cooled d is extracted with diethylammonium diethyl- ocarbamate in CCl_4, and the extract is heated an oxidising mixture of H_2SO_4, HNO_3 and O_4 (1:1:2), gently at first to remove CCl_4 and strongly to destroy remaining organic matter. cooled liquid is diluted, citric acid is added and pH is adjusted to 8·0 to 9·5 with aq. NH_3. he liquid, thermostatically controlled at 20°, yclohexanone oxalyldihydrazone soln. in aq. nol is added and, after development of the ur, the vol. is adjusted and the extinction is sured at 595 mμ and referred to a calibration h. Tin, Cd, Bi, Te, Ag, Zn and As, in amounts lly present, do not interfere. A. O. Jones

97. **Colour reaction for titanium.** F. S. Frum G. A. Kistanova. *Trudy Khim. i Khim. nol.*, 1958, (3), 578; *Ref. Zhur., Khim.*, 1960, Abstr. No. 69,166.—It is shown that the tion of 0·1% aq. 3-(*o*-hydroxyphenylazo)- motropic acid soln. to slightly acid Ti^{4+} soln. uces a blue or violet colour. The sensitivity ne reaction is 3 μg per ml; the limiting concn. n $3·3 \times 10^5$. The detection of Ti is not affected the presence of Mg^{2+} (1:700), Ca^{2+} (1:650), + (1:80), Zn^{2+} (1:20) and Al (1:2); Fe inter- . With a Ti concn. of 5 to 100 μg per ml the r of the colorimetric determination is ≃5%. K. R. Cook

1898. **Precipitation of titanium in the presence of Trilon B [EDTA].** V. P. Kopylova and T. N. Nazarchuk. *Ukr. Khim. Zhur.*, 1960, **26** (1), 110–112; *Ref. Zhur., Khim.*, 1960, (17), Abstr. No. 69,167.—Published results which indicate that Ti can be quant. pptd. by cupferron when Fe is masked with EDTA were tested. It was found that the separation of Ti and Fe was not possible under these conditions. K. R. Cook

1899. **Rapid titration of titanium in titanium slag.** Kimiyoshi Ono, Eiichi Hokoyama and Hiromoto Iwamatsu (Osaka Titanium Co., Higashihama, Amagasaki). *Japan Analyst*, 1959, **8** (12), 796–800. —The use of aluminium for the reduction of Ti^{IV} to Ti^{III} was examined. Up to 180 mg of TiO_2 is reduced to Ti^{III} in 3N to 4N-HCl with 1 g of aluminium without the use of an inert atmosphere. The reduced soln. is stable at temp. <50° in a flask closed with a Bunsen valve. No interference is caused by the presence of <0·5% of Cr, <0·25% of U and <2·5% of Sn in the sample. The standard deviation is 0·2% for samples containing 80 to 90% of TiO_2 and the determination takes 24 min. K. Saito

1900. **Determination of radio-zirconium.** G. J. Hunter and M. Perkins (U.K.A.E.A., Chem. Div., Woolwich). Analytical Method AERE-AM 67, 1960. 9 pp.—An analytical procedure is given for the separation of ^{95}Zr from HCl or HNO_3 soln. of fission products when the sodium content is <20 mg. Zirconium is isolated with zirconium carrier as $BaZrF_6$ after removal of rare-earth and alkaline-earth fission products by co-pptn. with LaF_3. The $BaZrF_6$ is dissolved in H_3BO_3 - HNO_3 and the Ba pptd. as the sulphate. The Zr is pptd. with aq. NH_3, the ppt. is dissolved in HCl and the Zr is converted into the mandelate and ignited to the oxide for γ-scintillation counting. Tables are given for calculating the activity. G. P. Mitchell

1901. **Chemistry of zirconium. VI. Method for the determination of zirconium in alloys.** J. Korkisch (Anal. Inst. Univ., Wien IX, Austria). *Z. anal. Chem.*, 1960, **176** (6), 403–405 (in German). —The sample (2 μg to 10 mg of Zr) is dissolved in mineral acid and evaporated to dryness. The residue is mixed with 6 to 10 parts of Na_2CO_3, fused in a platinum dish and treated as described previously for the determination of Zr in silicates (Korkisch and Farag, *Anal. Abstr.*, 1959, **6**, 3931). The Zr is separated by passing the soln. through a column of Dowex 1-X8; the zirconium - fluoride complex is retained. The Zr is then eluted with 4N-HCl and determined complexometrically or spectrophotometrically, with Solochrome violet R (C.I. Mordant Violet 5) as indicator or as colorimetric reagent. For the ion-exchange separation, ≯12 mg of Ti should be present. A. C. R. Hartley

1902. **Rapid determination of metallic zirconium and of oxygen in zirconium - oxygen alloys by the hydrogen evolution method.** M. E. Straumanis and T. Ejima (Dept. of Metall. Engng, School of Mines and Metall., Rolla, Mo., U.S.A.). *Z. anal. Chem.*, 1960, **177** (4), 241–244 (in English).—Dissolution of the sample (1 g) in HF (0·3 to 1N) and measurement of the H evolved (*cf.* Straumanis *et al.*, *Anal. Abstr.*, 1957, **4**, 1804) allows the amounts of free Zr and O present to be calculated. Other impurities present which do not develop H must be allowed for. The

time required is $\simeq$1 hr. and the reproducibility is $\pm$0·05%. J. W. PRICE

1903. **Determination of fluorine in zirconium metal by using a hollow-cathode discharge.** I. Korovin. *Zhur. Anal. Khim.*, 1960, **15** (5), 618–622. —Small amounts of F in zirconium metal are converted into ZrF_4 in a high-temp. discharge in a hollow cathode in an atmosphere of He and Ar with graphite electrodes. A band with a peak at 2274·5 A is produced, which is used to determine the F. A sensitivity of 3×10^{-4} % can be attained with a sample wt. of $\simeq$100 mg. Large amounts of alkali metals interfere, but other elements that form fluorides less stable than CaF_2 do not. J. W. PRICE

1904. **Organic azo-dyes in quantitative analysis. II. Selective complexometric indicator for thorium.** M. R. Zaki and K. Shakir (Atomic Energy Estab., Cairo). *Z. anal. Chem.*, 1960, **177** (3) 196–199 (in English).—Chrome red brown 5RD (C.I. Mordant Brown 41) (**I**) is a selective and sensitive indicator in the complexometric titration of micro and semi-micro amounts of Th^{4+} free from Zr and Fe. A study has been made of the variables that may cause errors in the determination, including the effect of concn. of Th^{4+} and dye, pH, dilution, temp. and foreign cations. An aliquot of the test soln. ($<$200 μg of Th per 10 ml), after separation from Fe and Zr, is diluted with acetic acid - acetate buffer (pH 2·5 to 3·5) to 10 ml, 1 ml of 0·1% aq. **I** soln. is added, and the mixture is titrated with EDTA (disodium salt) soln. The colour change at the end-point is from wine red to yellow. B. B. BAUMINGER

1905. **Detection of nitrogen in an inert gas.** A. I. Morozov. U.S.S.R. Pat. 128,195 (28th April, 1960).—Nitrogen is detected by the formation of the straw-yellow titanium nitride on a titanium-wire spiral heated to between 800° and 900° in the gas. The detection of 10^{-2} % (v/v) of N is possible. C. D. KOPKIN

1906. **Determination of nitrogen in metals by the combustion method. III. High-frequency combustion furnace.** Masahiko Tsuchiya (Inst. Tech.-anal. Chem., Fac. of Engng, Tokyo Univ. Hongo). *Japan Analyst*, 1959, **8** (11), 723–728.—The use of a high-frequency furnace enables a rapid combustion of the sample to be made in a current of O. When the evolved gas is extracted rapidly and foreign gases are removed, the total time taken for a determination is $<$60 min.

IV. Use of a high-frequency furnace for the determination of nitrogen. Masahiko Tsuchiya. *Ibid.*, 1959, **8** (11), 728–733.—When the content of N is $>$0·01%, the error is 4% for steel, titanium and ferrochromium and up to 10% for samples containing Si. Copper oxide is the best flux. The greatest source of error appears to be a large blank value, which results from rapid heating of the crucible. K. SAITO

1907. **Determination of nitrogen isotope abundance in ammonia-nitrogen.** R. Hüser, K. Habfast and M. von Bradke (Amalienstr. 52, München 13, Germany). *Z. anal. Chem.*, 1960, **176** (6), 429–436 (in German).—Nitrogen is liberated from NH_3 by the NaOBr reaction. The reactants are contained in the limbs of a special vessel (illustrated) which is evacuated to a known pressure. The reaction is then allowed to take place, and the evolved gas is passed into a mass spectrometer for measuremer ^{15}N. Errors due to fluctuations of the measu apparatus, and errors of measurement, the chem preparation of the sample, variations in the eq brium between $^{14}N^{14}N$ and $^{15}N^{15}N$ and the prese of foreign gases are discussed. The reproducibi ranges from 0·2 to 0·03%. A. C. R. HARTLE

1908. **Electrometric titration of hydrazine cerium(IV) sulphate.** P. V. Krishna Rao, Subramanyam and G. Gopala Rao (Chem. De Andhra Univ., Waltair, India). *Z. anal. Ch* 1960, **177** (1), 36–41 (in English).—Hydrazine H_2SO_4 soln. is titrated directly with Ce^{IV} at $\simeq$ in the presence of Mn^{2+} or Cr^{3+} as catalyst, wit consumption of 1·016 moles of $Ce(SO_4)_2$ per mol hydrazine. *Procedure*—The acidity of the soln adjusted to 0·5N to 1·0N with respect to H_2SO_4, $MnSO_4$ soln. (5 ml) is added and the soln. is dilu to 50 ml; the soln. is then titrated at $\simeq$50° v 0·1N- or 0·01N-$Ce(SO_4)_2$, with a platinum-ga indicator electrode and a saturated calomel refere electrode. Stable potentials are obtained 1 r after the addition of Ce^{IV}. A correction facto 0·985 is applied. The results show an error $\pm$0·2%. J. H. WATO

1909. **Quantitative analysis of phosphoric a IX. Differential spectrophotometric determina as molybdovanadophosphoric acid.** Masay Ishibashi and Masayuki Tabushi (Fac. of S Kyoto Univ., Sakyo-ku, Kyoto). *Japan Anal* 1959, **8** (12), 815–820.—By the use of a refere soln. containing 100 p.p.m. of P_2O_5, $\simeq$10 mg P_2O_5 can be determined. The colour reaches max. intensity 30 min. after the addition of reagents and is stable for 3 hr. By fuming w $HClO_4$, F^- are evaporated off and Si is conver into insol. SiO_2. *Procedure for ores*—Dissolve sample ($\simeq$0·4 g) in 5M-$HClO_4$ (10 ml) and 11N-H (2 ml) and heat to white fumes. Boil with wa (40 ml) for 3 min., filter and make up to 250 Make up an aliquot (20 ml) to 100 ml w 8M-$HClO_4$ (5 ml), 0·02M-NH_4VO_3 (10 ml), 0· $(NH_4)_2MoO_4$ (20 ml) and H_2O and measure extinction at 420 mμ, against the similarly trea reference soln. K. SAIT

1910. **Chemistry of condensed phosphates arsenates. XXVII. Separation on anion-excha resins and quantitative determination of conden phosphates having a degree of condensation to three.** W. Wieker (Inst. f. anorg. Che Dtsch. Akad. der Wissenschaften, Berlin). *Chem.*, 1960, **1** (1), 19–22.—Mixtures of conden phosphates have been successfully separated chromatography, under pressure, on columns Dowex 1-X10 and Wofatit SBW. The fracti were eluted with 0·25, 0·05 and 0·75M-KCl adjus to pH 4·5 with 10 ml per litre of buffer soln. (22· of Na acetate and 22·9 ml of acetic acid made to 500 ml). The phosphates in the eluates w determined colorimetrically after hydrolysis of condensed phosphates to the monophospha Identical mixtures were subjected to paper ch matography. Comparison of results shows t the column method gives more reliable figures, si the risk of hydrolytic decomposition is reduced. S. M. MARSH

1911. **New electrolyte for the polarograp determination of arsenic.** L. D. Dolaberidze a D. K. Kamkamidze. *Byul. Kavkaz. Inst. Mine*

ya, 1958 (1959), (2), 99–108; *Ref. Zhur., Khim.*,), (17), Abstr. No. 69,207.—It is shown that n 5N-NaOH, containing 40 g of mannitol per litre, he supporting electrolyte, As^{3+} and Sb^{3+} give inct anode waves with $E_{\frac{1}{2}} = -0{\cdot}29$ V and ·47 V, respectively, *vs.* the S.C.E. The linear tionship between the height of the wave and the cn. is maintained at $4{\cdot}6 \times 10^{-5}$ to 2×10^{-3}M As^{3+}, and at $5{\cdot}7 \times 10^{-5}$ to $1{\cdot}7 \times 10^{-3}$M for $^{+}$; As^{3+} can be determined in the presence of $^{+}$ if the ratio of Sb^{3+}: to As^{3+} is <10:1; with 0 mg of Sb^{3+} per 50 ml of soln. the results are satisfactory. Interference is given by Co^{2+}, $^{2+}$, large amounts of Fe^{2+}, SCN^-, and I^-.

K. R. Cook

912. **Photometric determination of antimony by iodic reduction to stibine.** G. Ciuhandu (Inst. Hygiene, Timişoara, Romania). *Rev. Chim., charest*, 1960, **11** (9), 530–532.—Antimony is uced in H_2SO_4 soln. at a lead cathode to SbH_3, l the gas is bubbled into an ice-cold alkaline n. of Ag *p*-sulphamoylbenzoate, which is selec-ely reduced by SbH_3 to a yellow-brown soln. riations in the light absorption, at 420 mμ, of n. containing 1 to 120 μg of Sb in 10 ml of 2N-SO_4 have been measured. H. Sher

913. **Determination of antimony in titanium sul-ate solution by alternating-current polarography.** mihiko Itsuki and Mitsuyoshi Nagao (Mitsubishi tal & Mining Co., Shinkawasaki-cho, Kita-ku, aka). *Japan Analyst*, 1959, **8** (12), 824–826.—e effect of acid concn. upon the a.c. polarography Sb is minimised in 2N-HCl containing 6N-H_2SO_4, l the influence of Ti (<4 g per 100 ml) and Fe^{II} 2 g) is negligible. The working curve is linear <2 mg of Sb per 100 ml. The error is 1·3%.

K. Saito

914. **Spectrochemical analysis of bismuth using hoto-electric spectrometer.** J. Forrest and H. L. ıston (Brookhaven Nat. Lab., Upton, New York, S.A.). *Appl. Spectroscopy*, 1960, **14** (5), 127–130. A rotating-disc solution method and a direct-ding spectrograph are used to analyse bismuth-ınium alloy for Mg (30 to 700 p.p.m.) and Zr) to 700 p.p.m.) with a precision of 2% of the ıtent, and Fe, Cr, Ni, Mo (10 to 500 p.p.m.) d Mn (5 to 250 p.p.m.) with a precision of 4 to % of content. Yttrium is added as an internal ndard. P. T. Beale

915. **Complexometric determination of bismuth d lead in bismuth-lead alloys.** V. F. Luk'yanov d L. I. Sedina. *Zhur. Anal. Khim.*, 1960, **15** , 595–597.—*Procedure*—Dissolve 0·1 g of sample 5 to 7 ml of HNO_3 (1:3), dilute to 60 ml, add g of urea and adjust the pH to 0·5 to 1·5 with . NH_3 (1:1). Titrate the Bi with 0·01M-EDTA sodium salt) to xylenol orange. Add 15 ml of a % aq. soln. of hexamine, bringing the pH to 4·5 5·5, and titrate the Pb. The accuracy of the ethod is high over the range 10 to 90% of Bi.

J. W. Price

916. **Polarographic determination of niobium in conium-base alloys.** D. P. Stricos (Knolls omic Power Lab., Schenectady, N.Y.). *U.S. omic Energy Comm.*, Rep. KAPL-M-DPS-3, 1960. pp.—A polarographic method was developed for e determination of Nb in zirconium-base alloys ntaining Sn. A sample was dissolved in a xture of H_2SO_4, HCl and HBF_4 and fumed with SO_4. The resulting H_2SO_4 soln. was diluted to a known vol., and the Nb was determined from its peak at −0·950 V *vs.* the S.C.E. using a cathode-ray polarograph. Nucl. Sci. Abstr.

1917. **Apparatus for determining a paramagnetic gas (oxygen) in a gas - air mixture.** V. A. Ferenets. U.S.S.R. Pat. 127,474 (25th March, 1960).—The apparatus comprises a working chamber containing a point or linear source of heat and a system of heat-sensitive elements, the latter being connected in a circuit containing a zero-indicator, the whole being in a magnetic field. The oxygen in the air passing through the chamber is deflected by thermal and thermomagnetic convection and changes the temp. of the heat-sensitive elements, so giving a reading on the meter, which is calibrated in % oxygen.

C. D. Kopkin

1918. **Detection of elementary sulphur by means of acetone.** G. Ingram and B. A. Toms (Courtaulds Ltd., Res. Lab., Maidenhead, Berks., England). *Analyst*, 1960, **85**, 766.—The test serves for the detection of elementary S in the presence of SCN^- or $S_2O_3^{2-}$ or both, but not in the presence of polysulphide. A drop of the test soln. is treated with one drop of Na_2S soln. (2% w/v) and then immediately with 0·5 ml of acetone. A blue or greenish-blue colour indicates the presence of elementary S. For solid samples a few mg of the powdered material is extracted with ethanol, with warming if necessary, and the Na_2S soln., followed by acetone, is added to the cooled extract. The lower limit of detection is 6 μg of S and the limiting dilution 1 in 3×10^4.

A. O. Jones

1919. **The chemistry of sulphur. LV. The analytical characterisation of elementary sulphur.** F. Fehér, K. H. Sauer and H. Monien (Chem. Inst., Univ. Cologne, Germany). *Z. anal. Chem.*, 1960, **177** (2), 109–113 (in German).—Four samples of sulphur from different sources have been examined for impurity content. By immersing a carbon-coated quartz heater at 750° in a bath of boiling sulphur (300 g) for 36 hr. all the impurities become adsorbed on the carbon (Wartenberg, *Z. anorg. Chem.*, 1956, **286**, 243). This technique has been used to prepare pure sulphur for the preparation of spectrographic standards, and also as a means of concentrating the impurities and providing a check on results obtained for sulphur. Results are reported for 10 elements semi-quantitatively and a further 16 qualitatively. T. R. Andrew

1920. **Separation of carrier-free sulphuric and phosphoric acids with anion-exchange resin.** Eiji Shikata and Chizuko Yamaguchi (Japan Atomic Energy Res. Inst., Tokai, Ibaraki-ken). *Japan Analyst*, 1959, **8** (11), 753–754.—When a mixture of carrier-free $H_3{}^{32}PO_4$ and $H_2{}^{35}SO_4$ in 0·5% NH_4Cl soln. is passed through a column (diam. 6 mm, length 100 mm) of Diaion SK-1 (100 to 200 mesh) (corresponding to Dowex 1) (La^{3+} or Fe^{3+} form), H_3PO_4 is quant. adsorbed, whilst only 5% of H_2SO_4 is adsorbed at pH 9. The H_3PO_4 is eluted with 2N-HCl. K. Saito

1921. **Redox indicators in the indirect volumetric determination of anions. Determination of sulphate ions with lead(II) nitrate.** Z. Gregorowicz and F. Buhl (Inst. for Inorg. Chem., Silesian Tech. High School, Gliwice, Poland). *Z. anal. Chem.*, 1960, **177** (2), 91–97 (in German).—After removal of potentially interfering bi- and ter-valent cations by pptn. as the carbonates, the soln., containing

2 to 100 mg of SO_4^{2-}, is neutralised to phenolphthalein with 0·1N-HNO_3, then heated, and an excess of $Pb(NO_3)_2$ soln. is added. After cooling the soln., methanol is added to a final concn. of 75% v/v, followed by 2 drops of $K_3Fe(CN)_6$ soln. (0·1N), and 2 drops of variamine blue soln. (1%). Excess of Pb^{II} is titrated with $K_4Fe(CN)_6$ soln. Results are within 1% of the theoretical.

T. R. Andrew

1922. **Indirect determination of sulphate ions by spectrophotometric titration of barium ions in the presence of barium sulphate.** Takuji Ito and Mitsuo Abe (Natural Resources Res. Inst., Tokyo Inst. Tech., Okayama, Meguro-ku, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1959, **62** (12), 1801–1805.—The effect of slit width, dilution and light scattering on the photometric titration of Ba^{2+} with EDTA was examined, with Eriochrome black T as indicator. When the dispersion of the suspension is kept constant by stirring in the presence of 0·01% of gelatin, and sufficient light (slit width 1·0 mm at 640 mμ) is passed through the titration cell, the relative error is <0·4% for the back-titration of Ba^{2+} ($\simeq$10 mg) in the presence of 4 to 100 mg of SO_4^{2-} per 10 ml. K. Saito

1923. **Chelatometric determination of sulphate ions as lead sulphate.** Tomoo Tanaka and Hiroshi Tanabe (Shizuoka Coll. of Pharm., Oshika, Shizuoka). *Japan Analyst*, 1959, **8** (12), 826–827.—Sulphate ions (<50 mg) are pptd. with 2% $Pb(NO_3)_2$ soln. in 30% ethanol, filtered off, washed with 25% ethanol and dissolved in 25% ammonium acetate soln. (<5 ml); the pH is adjusted to 5 with 20% acetic acid and the Pb is titrated with EDTA (disodium salt) (0·01M) with xylenol orange as indicator. The coeff. of variation is 2% for 1 mg, 0·5% for 10 mg, and <0·2% for 50 mg of SO_4^{2-}.
K. Saito

1924. **Sodium sulphite, hydrated (photographic grade).** British Standards Institution (2 Park Street, London, W.1). B.S. 3303:1960. 10 pp.—Tests for purity and a method of assay are given.

1925. **Sodium sulphite, anhydrous (photographic grade).** British Standards Institution (2 Park Street, London, W.1). B.S. 3304:1960. 10 pp.—Tests for purity and a method of assay are given.

1926. **Potassium metabisulphite (photographic grade).** British Standards Institution (2 Park Street, London, W.1). B.S. 3306:1960. 9 pp.—Tests for purity and a method of assay are given.

1927. **Ammonium thiosulphate solution (photographic grade).** British Standards Institution (2 Park Street, London, W.1). B.S. 3310:1960. 9 pp.—Tests for purity and content of thiosulphate are given.

1928. **Sodium thiosulphate, hydrated (photographic grade).** British Standards Institution (2 Park Street, London, W.1). B.S. 3301:1960. 9 pp.—Tests for purity and content of thiosulphate are given.

1929. **Sodium thiosulphate, anhydrous (photographic grade).** British Standards Institution (2 Park Street, London, W.1). B.S. 3302:1960. 9 pp.—Tests for purity and content of thiosulphate are given.

1930. **Determination of micro amounts of seleni in ores and rocks.** A. A. Sakharov (All-Union S Res. Inst. of Prospecting Methods and Tec Leningrad). *Zhur. Anal Khim.*, 1960, **15** (614–617.—*Procedure*—Treat 0·5 to 5 g of the fin ground material (containing >600 mg of Fe) w 25 ml of cold aqua regia and set aside overnig Filter, dilute to 150 to 200 ml, add 200 mg of Fe not present, partly neutralise with alkali and a an excess of an aq. suspension of ZnO. Heat coagulate the ppt., decant the supernatant liq and wash the ppt. by decantation with neut 0·1N-$ZnSO_4$. Centrifuge, dissolve the ppt. in t minimum amount of HCl, add 4 ml in excess a dilute to 10 ml. Heat to 70° to 80° and add so ascorbic acid to reduce the Fe and 500 mg in exce After 30 min. add $\simeq$200 mg of $(NH_4)_2SO_4$ and 1 of $BaCl_2$ soln. (10%); centrifuge, and wash the p with HCl (1:1) and H_2O. To the ppt. add 2 ml Na_2SO_3 soln. (10%) containing 25 g of Na_2CO_3 l litre and heat on a water bath for 10 min. Cent fuge and re-extract. To the combined soln. add equal vol. of HCl and heat for 50 min. Re-preci tate as described above, and centrifuge. Compare t red colour of the ppt. with that of standar Amounts of Fe >600 mg and Au interfere. In t determination of 2 to 30 μg of Se, the accuracy within 10 to 30%. J. W. Price

1931. **Determination of the form in whi selenium compounds are present in lead-producti dusts and sublimates.** V. V. Malakhov, I. Vasil'eva, E. I. Savichev, E. I. Golovin and E. Glotko (Leninogorsk Polymetallic Combine). *Zav Lab.*, 1960, **26** (9), 1060–1064.—Methods based dissolution in NaOH soln. to give the content selenites and selenates, treatment of the ins matter with Na_2SO_3 to dissolve the Se, and furth treatment of the insol. matter with HNO_3 (1:1) dissolve selenides of Pb, Zn and other metals, a described. G. S. Smith

1932. **Selenium and tellurium in meteorites.** DuFresne (Enrico Fermi Inst., Chicago Uni Chicago, U.S.A.). *Geochim. et Cosmoch. Acta*, 196 **20** (2), 141–148.—After decomposition of t sample with Na_2O_2, Se and Te are separated fr the bulk of the material by pptn. as the met with Au as a carrier: NaH_2PO_2 is a satisfacto pptg. agent. The ppt. is dissolved in HNO_3 a Se is determined spectrophotometrically on aliqu portions with 3,3′-diaminobenzidine and, after t separation of most of the Au by extraction wi ethyl ether, Te is determined by the iodotelluri method. The values obtained on samples wi known amounts of Se and Te added are general $\simeq$ 3% and 10% low, respectively.
R. A. Howie

1933. **Paper-chromatographic analysis in conne tion with the formation of tellurium(VI) compoun** A. Schneer and M. Ördögh (Cent. Res. Inst. f Physics, Budapest). *J. Chromatography*, 1960, (4), 319–322 (in English).—Compounds of Te^{VI} a Te^{IV} can be readily detected in the presence each other by chromatography on S. & S. 204 paper with n-butanol saturated with 3M-HCl, the ascending technique. The spots are located spraying the chromatogram with $SnCl_2$ soln. T R_F value of Te^{VI} is 0·08 to 0·15; that of Te decreases with increase in pH, but in neutral

soln. it is 0·51 to 0·61. The minimum detect- amounts are 2 μg of Te^{VI} and 1 μg of Te^{IV}, each ne presence of a large excess of the other.
R. M. Rowley

934. **Complexometry of tervalent chromium. I. omic chloride.** N. N. Pavlov, A. R. Kuznetsov G. A. Arbuzov. *Izv. Výssh. Uchebn. Zavedenií, hnol. Legk. Prom.*, 1960, (1), 54–59; *Ref. Zhur., m.*, 1960, (18), Abstr. No. 73,075.—The deter- ation of Cr^{3+} by boiling with EDTA (disodium (**I**) with subsequent titration of the excess of **I** $NiSO_4$ soln. [*Ref. Zhur., Khim.*, 1958, (1), tr. No. 892] is modified in some details.
C. D. Kopkin

935. **The absolute abundance of the chromium opes in chromite.** G. D. Flesch, H. J. Svec and G Staley (Atomic Res. Inst., Iowa State Univ. Sci. and Technol., Ames, U.S.A.). *Geochim. et moch. Acta*, 1960, **20** (3–4), 300–309.—The high our pressure of CrO_2F_2 makes it ideal as a means ntroducing Cr into the mass spectrometer as a giving extremely stable sample conditions. To id the considerable chemical manipulation uired to convert chromite ores into CrO_3, to give $_2F_2$, a method has been devised in which CrO_3 eplaced by $PbCrO_4$, which is readily separated n the leachings of chromite - sodium peroxide ons. Full details are given of the fusion and hing processes and of the reaction of the $PbCrO_4$ h CoF_3 to produce CrO_2F_2. The mass-spectro- phic techniques are outlined. R. A. Howie

936. **Volumetric determination of molybdenum ferromolybdenum.** S. M. Gubel'bank, V. I. rbatova and E. V. Silaeva. *Byull. Nauch.- h. Inform. Ural'sk. Nauch.-Issled. Inst. Chern. tallov*, 1959, (7), 127–130; *Ref. Zhur., Khim.*, 0, (18), Abstr. No. 73,077.—The method is based the reduction of Mo^{VI} to Mo^{V} in a bismuth uctor, with subsequent titration of the Mo^{V} h $KMnO_4$ soln. The method has been tested on ndard ferromolybdenum samples with contents Mo of 60·28 and 57·01%; the standard deviation s ±0·11%.
C. D. Kopkin

937. **Use of solid electrodes for the amperometry molybdenum and tungsten.** S. K. Chirkov and S. Studenskaya. *Byull. Nauch.-Tekh. Inform. al'sk. Nauch.-Issled. Inst. Chern. Metallov*, 1959, 113–120; *Ref. Zhur., Khim.*, 1960, (18), Abstr. 73,080.—A method is developed for the ampero- tric titration of WO_4^{2-} and MoO_4^{2-} with $Pb(NO_3)_2$ n. in acetate buffer soln. (pH 4 to 5) by the dation current of Pb^{2+} (at 1·2 to 1·5 V), with identical platinum wires as electrodes. The n. is stirred by rotating the beaker. Ions reduced a platinum cathode (*e.g.*, Cu^{2+}, VO_3^-, $Cr_2O_7^{2-}$) not interfere, but ions oxidised on a platinum ode (*e.g.*, Fe^{2+} and Cr^{3+}) interfere. When Mo and are both present, the W is removed by pptn. as gstic acid. The method is applicable to the ermination of Mo in ferromolybdenum, other n alloys and steel, and to the determination of in steel.
C. D. Kopkin

938. **Polarographic determination of tungsten.** S. Deshmukh and J. P. Srivastava (Benares du Univ., Varanasi, India). *Zhur. Anal. Khim.*, 60, **15** (5), 601–604.—In a soln. 0·2M in Na late containing 0·005% of gelatin and at pH 0·5 l, W^{VI} gives a wave with $E_{\frac{1}{2}} = -0{\cdot}62$ V. In s medium, SO_4^{2-}, HSO_4^-, Cl^-, alkali metals, Cu, Ni, Mn and Cr do not interfere, but NO_3^- and V interfere and must be absent; Fe^{III} and Mo^{VI} can be determined in the same soln.
J. W. Price

1939. **Spectrophotometric studies of organometallic complexes used in analytical chemistry. VII. Spectrophotometric determination of uranium with 3-hydroxyflavone.** Takuji Kanno (Res. Inst. Mineral Dressing and Metallurgy, Tohoku Univ., Nagamachi, Sendai). *Japan Analyst*, 1959, **8** (11), 714–717.—3-Hydroxyflavone (**I**) forms a yellow complex with U, which is extracted with tri-n-butyl phosphate (**II**) at pH 6·0 to 7·0. The organic layer gives max. absorption at 400 to 450 mμ and the extinction is proportional to the concn. of U (<12 μg per ml) in the presence of $1{\cdot}3 \times 10^{-3}$M-**I**. A mixture (1 : 1) of **II** and hexane is also suitable as extractant; U reacts with **I** in the mixture, after being extracted with the same mixture from 0·02M-HNO_3 containing 30% of NH_4NO_3.

VIII. Composition and formation constant of the uranyl - 3-hydroxyflavone complex. Takuji Kanno. *Ibid.*, 1959, **8** (11), 718–722.—Two kinds of complex are formed, the molar ratio of U to **I** being 1 : 1 and 1 : 2 in 60% acetone and the over-all formation constant is 2×10^{19}. In **II**, the 1 : 2 complex, which is useful for chemical analysis, appears to contain 1 mole of co-ordinated **II**.
K. Saito

1940. **Polarographic determination of uranium in the presence of plutonium.** M. E. Smith (Los Alamos Sci. Lab., N. Mex.). *U.S. Atomic Energy Comm.*, Rep. LA-1249, 1956. 30 pp.—A polarographic method for the determination of U in the presence of Fe and Pu is described. 2M-Hydroxyammonium chloride is used as the supporting electrolyte. This solution reduces the Pu to Pu^{3+} without reducing the U. The U is determined polarographically by the difference in uranyl reduction wave height (U^{VI} to U^{V}) at −0·35 V. Solutions with a U to Pu weight ratio as low as $3{\cdot}5 \times 10^{-3}$ were analysed with an error <2%.
Nucl. Sci. Abstr.

1941. **Titrimetric determination of uranium [iron and vanadium] in presence of organic solvents.** U. Veereswara Rao (Inst. of Nuclear Sci. "Boris Kidrich", Belgrade, Yugoslavia). *Z. anal. Chem.*, 1960, **177** (3), 190–195 (in English).—The conditions for this determination have been studied and it is shown that the determination of U and Fe^{III} by reduction in a cadmium reductor, and subsequent oxidation by titration with vanadate, can be carried out in the presence of org. solvents provided that during the titration the acid concn. is molar. The soln. containing the reduced U^{4+} or Fe^{2+} is made molar in acid and titrated with 0·1N-$NaVO_3$ (**I**), with diphenylbenzidine as indicator. To obtain a better colour change towards the end-point, 2 ml of 10% oxalic acid soln. is added. A similar procedure is applicable to the determination of V^{V}. A known excess of 0·1N-$FeSO_4$ is added to the test soln., and the unchanged $FeSO_4$ is determined by titration with **I**.
B. B. Bauminger

1942. **Absorptiometric determination of uranium in grass.** U.K.A.E.A., Chemical and Metallurgical Services (Springfields, Lancs.). U.K.A.E.A. Report PG 142(S), 1960. 10 pp.—After wet oxidation with H_2SO_4 - HNO_3, SiO_2 is removed with HF. The residue is dissolved in HNO_3 and the U is co-pptd. with $Fe(OH)_3$. The ppt. is re-dissolved in HNO_3 and the soln. extracted with ethyl ether. The soln.

is evaporated to dryness, the residue is dissolved in HNO_3 and again extracted with ethyl ether. The soln. is evaporated to dryness with HNO_3 and $HClO_4$. The residue is dissolved in dil. H_2SO_4, neutralised with aq. NaOH soln. and poured into a soln. of NaOH and $Na_4P_2O_7$. The U is determined absorptiometrically with H_2O_2. N. E.

1943. **Spectrographic determination of boron in metallic uranium and uranium tetrafluoride.** Shizo Hirano, Hitoshi Kamada and Takao Nishiya (Inst. Tech.-anal. Chem., Fac. of Engng, Tokyo Univ., Hongo, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1959, **62** (5), 622–625.—The procedure of Coursier *et al.* for the extraction of BF_4^- with tetraphenylarsonium chloride (**I**) (*Anal. Abstr.*, 1956, **3**, 661) was applied to the spectrographic determination of B in uranium (terminal voltage, 280 V, preliminary discharge, 5 amp. for 12 sec.; exposure, 15 amp. for 60 sec.). Graphite is the best spectrographic buffer and Ir is used as the internal standard. The intensity of the lines of B increases with increase in current, becoming constant at 15 amp. The working curve (B 2497·73 *vs.* Ir 2502·98) is linear for $<0{\cdot}3$ μg of B. The formation of BF_4^- (<1 μg of B) is complete within 10 hr. at room temp. but is more rapid at 170° to 180°. *Procedure for the extraction of B*—Heat the sample (0·5 to 1 g) with a mixture (1 : 1) of H_3PO_4 and 10% HF (5 to 10 ml) with addition of 2N-HF (10 to 15 ml). Add HNO_3 (1 : 3) until U is converted into the sexavalent state, cool and dilute to 40 ml with 2N-HF (5 ml) and water. Shake with a soln. of **I** in $CHCl_3$ (0·4%) (10 ml) for 30 min., evaporate the org. layer to dryness with 0·1N-NaOH (15 drops) and mix the residue with graphite (0·1 g) containing 1 part in 10^4 of iridium powder for spectrographic examination. K. Saito

1944. **Spectrophotometric determination of micro amounts of nitrogen in uranium and zirconium.** Tamotsu Tanaka and Tsugio Takeuchi (Inst. Tech.-anal. Chem., Fac. of Engng, Nagoya Univ., Chikusa-ku, Nagoya). *Japan Analyst*, 1959, **8** (12), 822–824.—Kruse and Mellon's pyridine - pyrazolone method (*Anal. Abstr.*, 1954, **1**, 276) was applied to metallic uranium and zirconium. The sample (0·2 g) is dissolved in HCl (for uranium), or in HCl and HF (for zirconium) and the NH_3 is distilled into a separating-funnel. The distillate is adjusted to pH 3·7, treated with chloramine T and 3-methyl-1-phenyl-5-pyrazolone in pyridine (0·1%), and extracted with CCl_4. The extinction is measured at 450 mμ. K. Saito

1945. **Determination of rare earths in uranium dioxide.** U.K.A.E.A. (Res. Group, Chatham Outstation, H.M. Gun Wharf, Chatham, Kent). Analytical Method AERE-AM 62, 1960. 24 pp.—Full details are given of a procedure in which the rare-earth metals are separated on a cellulose column from a soln. of uranium dioxide (5 g). The column is ashed and, after the co-pptn. of the rare-earth metals with Ca oxalate and then with $Fe(OH)_3$, they are determined spectrographically. The method is suitable for the analysis of material containing down to 1 p.p.m. of La, Eu, Cd, Tb, Dy and Yb and down to 2 p.p.m. of the other rare-earth metals. The error of the mean of duplicate determinations should be $<20\%$. G. J. Hunter

1946. **Complexometric determination of plutonium (IV) with arsenazo indicator.** P. N. Paleĭ and Ven-Tsin Chzhan (V. I. Vernadsky Inst. of Geochem. and Anal. Chem., Acad. of Sci., USSR, Mosco *Zhur. Anal. Khim.*, 1960, **15** (5), 598–60 *Procedure*—To a soln. of 2 to 20 mg of Pu^{IV} 40 to 100 ml of HCl or HNO_3 (0·1 to 0·2N) 10 to 20 drops of a 0·4% aq. soln. of arsenazo titrate with 0·005M to 0·01M-EDTA to the col change from blue - violet to rose. There is interference from La^{3+}, UO_2^{2+}, Cr^{3+}, Pb^{2+} or N but the concn. of Fe^{3+} must not be greater t 4% of the Pu. J. W. Pric

1947. **Chemical analysis of binary alloys plutonium with aluminium, iron or uraniu** D. H. F. Atkins and E. N. Jenkins (Chem. D A.E.R.E., Harwell, England). A.E.R.E. Rep AERE-C/R 2161, 1960. 22 pp.—The alloys dissolved in HCl and, after making the soln. with respect to acid by adding HNO_3, and ensur that the Pu is in the Pu^{IV} state, it is passed throu a column of De-Acidite FF, which retains only Pu. Standard methods are used to determine elements in the percolate. The Pu is eluted fr the resin with M-HNO_3 and 5% (w/v) hydro amonium chloride soln. and determined by dif ential absorptiometry of the Pu^{3+}. This proced can also be used to determine Pu in soln. contain up to at least 9 times as much U (w/w) and 2 times as much Al. Full experimental details of procedures are given. G. J. Hunte

1948. **Photometric determination of fluorine mica by the ferric thiocyanate method.** Nob Shimoda and Naomichi Kuriyama (Chem. La Hokkaido Gakugei Univ., Kushiro). *Japan Anal* 1959, **8** (11), 743–745.—Since the CaF_2 method unsuitable for $<1\%$ of F in silicate rocks, thiocyanate method (Okuno, *J. Chem. Soc. Jap* 1941, **62**, 1158; Ingols *et al.*, *Anal. Chem.*, 1950, 799) was applied. The error was then within for 1 mg of F^-, within 3% for 0·7 mg, within for 0·3 mg and within 20% for 0·1 mg per 100 The colour is stable for 4 hr. in the presence of 30 of NH_4SCN and 2 mg of Fe^{III}. K. Saito

1949. **3-Hydroxy-3-phenyl-1-*p*-sulphophen triazen as an indirect spectrophotometric reagent fluoride.** S. Mehra and N. C. Sogani (Gov. Co Ajmer, India). *J. Indian Chem. Soc.*, 1960, **37** 562–564 (in English).—Fluoride ion (0·025 to 2 m can be determined colorimetrically by its bleachi effect on the complex of Fe^{III} with 3-hydroxy phenyl-1-*p*-sulphophenyltriazen (**I**) (Gupta a Sogani, *Anal. Abstr.*, 1961, **8**, 568). The sam soln. (25 ml) is buffered to pH 3 with 10% acetate soln. and 10% HCl, 0·001M-$FeCl_3$ (10 and 0·4% **I** soln. (10 ml) are added and the extinct is measured at 650 mμ. Ions that form sta complexes with Fe^{3+} or F^- interfere. W. T. Carte

1950. **Potassium bromide (photographic grad** British Standards Institution (2 Park Stre London, W.1). B.S. 3307 : 1960. 12 pp.—Tests purity and a method of assay are given.

1951. **Determination of potassium iodide sodium chloride.** L. N. Lapin. *Izv. Ak Nauk UzSSR, Ser. Med.*, 1959, (6), 60–65; *Zhur., Khim.*, 1960, (17), Abstr. No. 69,248 *Procedure*—Dissolve the sample (50 g) in H (≃200 ml), add CaO (0·5 g) to precipitate Fe(OH dilute to 250 ml and filter off the ppt. Add H (1 : 1) (1 ml) and 0·5% $NaNO_2$ soln. (0·5 ml) to filtrate (5 to 20 ml) and 5 min. later add brilli

n soln. (0·5 g in 25 ml of ethanol plus 75 ml of (0·5 ml), shake, add toluene (10 ml) and shake n for ≃30 sec; after a further 30 sec. separate the nic layer and compare its colour against dards containing 4, 8, 12, 20, 30 40, 80 and g of KI. Nitrates of Cu and Ni are recom-ded as standards in place of KI because of their lity. A single determination takes <10 min.

K. R. Cook

52. **Rapid reductimetric determination of manse in alloys and minerals.** I. Kluh, J. Doležal J. Zyká (Inst. f. Anal. Chem., Charles' Univ., ue, Czechoslovakia). *Z. anal. Chem.*, 1960, (1), 14–20 (in German).—The oxidation of Mn^{II} Mn^{III} with $HClO_4$ (Ingamells and Bradshaw, *. Abstr.*, 1958, **5**, 4114) leads to low results. ntitative results are obtained by oxidation with O_3 in the presence of excess of K^+. *Procedure*— soln. of Mn (≃16 mg) is treated with 20 ml of O_4 (80%) and with 1 to 3 g of K_2CO_3. Potas-bromate (0·1 g) is added and the soln. is ed until no more fumes of Br are evolved and for a further 4 min. After cooling and diluting ≃50 ml, the soln. is titrated with 0·05N-$_4)_2SO_4.FeSO_4$ or 0·05N-quinol. The end-point etected potentiometrically, or with diphenyl-ne, added just before the end-point. The lts show a mean error of ±0·4%. Methods are n for the determination of Mn in ferroman-se, manganese ore and manganese bronze.

J. H. Waton

53. **Rapid successive determination of iron(III) aluminium in manganese ore with EDTA and TA.** Yoshihide Endo and Hajime Takagi iai Plant, Kawasaki Iron & Steel Co-op., iai-ku, Kobe). *Japan Analyst*, 1959, **8** (12), -830.—When Fe^{III} is titrated with EDTA dium salt) at pH 2 to 3 with salicylic acid as cator, and Al is then titrated with CyDTA diaminocyclohexane-*NNN'N'*-tetra-acetic acid) pH 2·2 with Cu - PAN [1-(2-pyridylazo)-2-nthol] as indicator, a large amount of Mn does interfere. Alumina (1 to 6%) and Fe_2O_3 (2 to ,) in manganese ore can be determined within nin.

K. Saito

54. **Radiochemical determination of technetium-in nitrate solutions by solvent extraction of aphenylarsonium pertechnetate.** U.K.A.E.A., luction Group (Windscale, Cumberland, Eng-). U.K.A.E.A. Report PG 161 (W), 1960. pp.—Full details are given of a procedure icable to soln. of mixed fission products. nnetium sulphide is co-pptd. with PtS by H_2S a soln. containing hydroxyammonium chloride. ppt. are dissolved in aq. NH_3 - H_2O_2, and, after addition of tetraphenylarsonium chloride and nonium carbonate soln., tetraphenylarsonium echnetate is extracted into $CHCl_3$. The extract ashed with NaOH soln. and evaporated. The tivity of the residue is measured both with and out an added absorber, so that a correction be made for any radio-ruthenium in the ce. The coefficient of variation in 30 deter-ations by three operators was 8%. Nitrate ion n amount >40 millimoles interferes.

G. J. Hunter

55. **Extraction - photometric determination of es of iron with 1-nitroso-2-naphthol.** A. B. nk and A. M. Bulgakova (All-Union Sci. Res. . of Chem. Reagents, Karkov Branch). *Zhur. Anal. Khim.*, 1960, **15** (5), 605–609.—The green complex formed from Fe^{2+} and 1-nitroso-2-naphthol (**I**) can be used for the determination of Fe in materials of high purity. Moderate amounts of Cu, Co and Ni do not interfere; Ag interferes in large amounts, but Al, Cd, Mn, Tl, Zn, PO_4^{3-}, citrate and tartrate can be present in excess. *Procedure with sodium iodide*—Dissolve 10 g of sample in 0·05M-ammonium tartrate (25 ml) containing 0·5 ml of $Na_2S_2O_3$ soln. (3%), add 10% ascorbic acid soln. (15 ml), neutralise with aq. NH_3 and add 5 ml of **I** soln. (dissolve 1 g in 120 ml of N-KOH and dilute to 1 litre). Dilute to 150 ml and after 40 min. extract twice (15, 10 ml) with isopentyl alcohol previously saturated with CO_2. Combine the extracts, make up to 25 ml and measure the extinction, with a red filter. As little as 5×10^{-6}% of Fe can be determined. A similar procedure is used with a 5-g sample of tartaric acid.

J. W. Price

1956. **Determination of iron by extraction with ethyl acetoacetate.** S. E. Kreĭmer, A. V. Stogova and A. S. Lomekhov. *Zavod. Lab.*, 1960, **26** (10), 1104–1106.—To determine Fe in cathodic nickel, the sample (1 g) is dissolved in 25 ml of HNO_3 (3:2), the soln. is evaporated down to a small volume and cooled, and the free acid is neutralised by addition of 25% Na acetate soln., of which ≯0·5 to 1 ml should be required. The soln. at a pH of 5 is treated with 30 ml of an acetate buffer soln. of pH 5·57 and then diluted with water to a volume of 100 ml. Ethyl acetoacetate (2 ml) and 10 ml of $CHCl_3$ are added and the mixture is shaken in a separating-funnel for 5 min. The separated aq. layer is washed with $CHCl_3$, and the extinction of the extract and washings after dilution to 25 ml with $CHCl_3$ is measured at 450 mμ. The method is applicable also to the determination of Fe in cobalt. Interference due to the presence of Cu can be prevented by addition of NH_4SCN. Satisfactory results were obtained over the concn. range 0·004 to 0·4%.

G. S. Smith

1957. **Spectrophotometric determination of iron(III) with acetylacetone.** Tsunenobu Shigematsu and Masayusi Tabushi (Inst. Chem. Res., Kyoto Univ., Sakyo-ku). *Japan Analyst*, 1959, **8** (11), 710–714.—The absorption spectrum of Fe^{III} acetylacetonate is influenced by pH, but remains unchanged at pH 5 to 8·5. The extinction is proportional to the concn. of Fe for <10 p.p.m. at 350, 355 and 440 mμ at pH 7 in the presence of 0·3 to 0·5% of acetylacetone. There is no interference from Al, Ag, Cd, Mg, Mo, Pd, W or Zn (<10 mg each); Cu, Co, Mn and Ni increase the extinction.

K. Saito

1958. **Determination [of tervalent iron] with ferrocene.** L. Wolf, H. Franz and H. Hennig (Inst. f. anorg. Chem., Karl-Marx-Univ., Leipzig). *Z. Chem.*, 1960, **1** (1), 27–28.—The method is based on the reaction of Fe^{3+} with unsubstituted ferrocene (dicyclopentadienyliron). The sample soln., containing ≃10 mg of Fe^{3+} in 200 to 250 ml, is treated with 5 ml of N-NH_4SCN, 10 ml of HCl and a few drops of dil. ethanolic soln. of 2,4-dinitro-*N*-phenylpyridinium chloride before titration with 0·01N-ethanolic ferrocene soln. At the end-point the colour change is from red to blue. Amounts of 5 mg of Fe can be determined to within ±0·025 mg. Large excesses of Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Zn^{2+}, Cd^{2+}, Hg^{2+}, As^{3+}, Sb^{3+}, Bi^{3+}, Al^{3+}, Cr^{3+}, Ti^{4+}, and alkali and alkaline-earth metals do not interfere,

but Cu^{2+} and H_2O_2 do interfere. The effect of F^- can be masked by adding $ZrOCl_2$ before the reaction. The method is particularly applicable to the analysis of ferrocenoylacetone and 1,1′-bis(acetoacetyl)-ferrocene. The reaction can also be applied to the determination of Co^{2+}. S. M. MARSH

1959. **Rapid polarographic determination of iron in alkali hydroxide.** Toshihida Saito and Isao Muraki (Osaka Ind. Res. Inst., Ooyodo-ku). *Japan Analyst*, 1959, **8** (11), 707–710.—Tervalent Fe is completely converted into its mannitol (**I**) complex within 1 hr. at 90° in 0·1M to 0·3M-**I**. The $E_{\frac{1}{2}}$ value $= -1{\cdot}09$ V and $-1{\cdot}58$ V *v.s.* the S.C.E. in 0·1M-**I** in 3N-NaOH and the wave height is proportional to the concn. of Fe for <30 μg per ml. *Procedure*—Dissolve the sample of alkali hydroxide and **I** in water to produce a soln. of the required concn., heat at 90° for 1 hr., add gelatin (0·0005%), pass N, and record the polarogram. K. SAITO

1960. **Chromatographic separation of ferric ions from zinc or nickel ions.** I. A. Korshunov and A. I. Subbotina. *Trudy Khim. i Khim. Tekhnol.*, 1959, (1), 55–57; *Ref. Zhur., Khim.*, 1960, (17), Abstr. No. 69,113.—The method is based on the differences in stability of the oxalate complexes of Fe^{3+} and Zn^{2+} and of Fe^{3+} and Ni^{2+}; the cation-exchange resin KU-2 is used and adsorption takes place from soln. with salt concn. of 0·02 to 0·05 mole per ml at pH 1·5 to 2·5; Fe^{3+} are eluted with 0·05M-oxalic acid, containing 0·1% of H_2O_2 to prevent reduction of Fe^{3+}; Zn^{2+} or Ni^{2+} are eluted with 2N-HCl. Nickel is determined by titration with EDTA in ammoniacal medium in the presence of murexide; Zn^{2+} or Fe^{3+} are determined by the use of the radioactive indicators ^{65}Zn and ^{59}Fe, respectively. K. R. COOK

1961. **Combined method of analysis for high-alloy iron and steel.** W. B. Sobers (Chain Belt Co., Milwaukee, Wis., U.S.A.). *Foundry*, 1960, 110–113.—Details are given of a rapid composite scheme for the analysis of seven elements in high-alloy iron and steel. A 1-g sample is dissolved in HNO_3 - HCl and fumed with $HClO_4$. Silicon is determined gravimetrically, the soln. being then made up to 200 ml. Aliquots of this are used for the colorimetric determinations of Mn as permanganate, Cr as the diphenylcarbazide, P as molybdophosphate, Ni as the dimethylglyoxime complex, Mo as the thiocyanate and Cu as the diethyldithiocarbamate complex. G. P. MITCHELL

1962. **New method for the carbide analysis of steel.** L. Brháček, A. Golonka and K. Kurzová (Výzkumný ústav VŽKG, Ostrava, Czechoslovakia). *Hutn. Listy*, 1960, **15** (9), 679–686.—Special features of the method are the short time of electrolysis for isolating the carbides, in order to reduce decomposition, and a new method of chemical analysis, said to give better results than the dry method. After chemical polishing, the cylindrical test piece is weighed and electrolysed in, *e.g.*, a citrate electrolyte, with a current density of 1 to 2 amp. per sq. cm for 1 to 2 min. The carbides are brought into suspension in water by means of ultrasonic vibrations and the test piece is re-weighed; after dissolution with H_2SO_4 (1:9), the sum of Mn, Cr and V is determined by amperometric titration with $(NH_4)_2SO_4.FeSO_4$, after the addition of $AgNO_3$ and $(NH_4)_2S_2O_8$. More $(NH_4)_2S_2O_8$ and conc. HCl are then added, and the soln. is warmed till the pink colour is discharged, then titrated again. This result represents Cr plus V. An excess $(NH_4)_2SO_4.FeSO_4$ is added and V is determined adding $KMnO_4$ and $NaNO_2$ till no change in tinction is observed, then titrating with $(NH_4)_2SO$ $FeSO_4$. Manganese can be determined pho metrically in the original soln. by KIO_4 oxidat in HNO_3 soln. Iron is determined with 1,10-phen throline and hydroxylamine in the presence of acetate. For Mo, NH_4SCN and $SnCl_2$ are used. P. GLOVER

1963. **Determination of trace amounts of tantal in steel.** L. Kidman, C. L. Darwent and G. Wh (English Steel Corp. Ltd., Sheffield). *Metallurg Manchr*, 1960, **62**, 125–128, 130.—A sim absorptiometric procedure is described. The Ta separated from Ti by double pptn. from HCl so with benzenearsonic acid, with Zr as carrier. T Ta is determined absorptiometrically at 26° as pyrogallol complex. The complex formed is sta provided that the temp. is maintained consta The effect of Nb, Zr, Mo, W, V and Ti on the co plex was examined; Zr did not interfere and M W and V were satisfactorily removed by precipitation of the Ta. Experiments showed th when Zr is used as carrier a quant. pptn. of occurs and a complete separation from Ti is effect The colour of the Nb - pyrogallol complex is a minimum at pH 2, at which the Ta is determin By varying the pH, Nb and Ta can be determin independently in the same sample. M. G. SEAMAN

1964. **Photometric determination of titanium steel] as a pyridine - salicylate complex.** A. Babko and A. I. Volkova (Inst. of Gen. and Ino Chem., Acad. of Sci., Ukrainian SSR, Kiev). *Zh Anal. Khim.*, 1960, **15** (5), 587–590.—*Procedur* Dissolve 0·1 to 0·2 g (containing 0·1 to 1 mg of in H_2SO_4 (1:9) and HNO_3; boil the soln. to remo oxides of N, then cool, and make up to 50 100 ml. Adjust a 10-ml aliquot with aq. NH_3 (1 to pH 1 to 2, add 2 drops of NH_4SCN soln. (1%) a $Na_2S_2O_3$ soln. (10%) dropwise until the red colo is discharged. Add 2 ml of Na salicylate so (10%) and 0·1 g of pyridine [or quinoline or 5 ml amidopyrine soln. (2%)], adjust to pH 3·5 with NH_3 and add acetate buffer soln. (pH 3 to 3·5) ml). Extract with 5 ml of $CHCl_3$ ($\times$ 4) and make the combined extracts to 25 ml. Measure extinction at 400 to 430 mμ. The sensitivity of method is 10 μg of Ti in 25 ml of extract. J. W. PRICE

1965. **The difference between analytical resu for nitrogen [in steel] by the vacuum fusion meth and acid distillation method.** Mutsumi Ih (Kawasaki Iron Works, Nippon Steel Tube C Minami-watarida-cho, Kawasaki). *Japan Analy* 1959, **8** (12), 786–791.—The acid distillation meth gives higher results than does the vacuum fus method for low-carbon steel and low-carbon ferr alloys. In molten iron containing Cr, a sm amount of nitrogen is retained at 10^{-4} to 10^{-5} to Vacuum-evaporated aluminium (or other meta on the inside wall of the apparatus absorbs significant amount of N. K. SAITO

1966. **Polarographic determination of arsenic iron, steel and iron ore.** Hiroshi Asaoka (Fac. Commerce, Hitotsubashi Univ., Kunitachi, Toky *Japan Analyst*, 1959, **8** (12), 774–778.—Arse ($>0{\cdot}01\%$) in iron is co-pptd. with MnO_2 in 0· HNO_3, distilled in a current of CO_2 into a tarta

d (**I**) soln. and determined polarographically. e working curve is linear for <1 mg of As per) ml, in M-**I** containing 1·5N-HCl and 0·004% gelatin; Sn and Sb affect the polarogram but are t distilled under the specified conditions. *Pro- ure*—Dissolve the sample (1 g) in HNO_3 (1:1) ml) or aqua regia, dilute to 100 ml, boil, add $(NO_3)_2$ soln. (5%) (5 ml) and $KMnO_4$ soln. (2%) ml) and filter. Transfer most of the MnO_2 to flask, dissolve the remainder in H_2SO_4 (1:29)) ml) and H_2O_2 (30%) (a few drops). Heat both rtions to white fumes, add conc. HCl (20 ml) and drazine sulphate (0·5 g) and distil, collecting 10 ml within 15 min. in 50 ml of water, and dilute to 100 ml in a flask containing **I** (15 g). Add latin soln. (0·2%) (2 ml). Measure the wave height −0·33 V *vs.* the S.C.E. K. Saito

1967. **Determination of vanadium in steel and rro-alloys by amperometric titration with two dicator electrodes.** L. S. Studenskaya and O. A. ngina (Ural Sci. Res. Inst. of Ferrous Metals). *vod. Lab.*, 1960, **26** (10), 1102–1104.—The sample to 2 g) is dissolved in 50 ml of H_2SO_4 (1:4) and idised with 10 ml of conc. HNO_3. The soln. is aporated to fuming and the residue is dissolved 30 to 50 ml of water. The cooled soln. is treated th 0·05N-$KMnO_4$ until it turns pink, followed mediately by one or two drops of 0·2% $NaNO_2$ ln. and then by 0·2 to 0·5 g of urea. Two platinum ectrodes together with a stirrer are introduced, d the V is titrated at an applied potential of 5 to 0·6 V with $FeSO_4$ soln. Alternatively, the ration can be carried out without first destroying e excess of $KMnO_4$. The amperometric titration rve then shows three sections, the middle hori- ntal section corresponding to the content of V. G. S. Smith

1968. **Radiochemical investigation of the com- stion method for the determination of sulphur in st iron.** R. C. Rooney and F. Scott (B.C.I.R.A., lvechurch, Birmingham). *J. Iron St. Inst.*, 1960, **95** (4), 417–421.—A detailed description is given r the determination of S with ^{35}S as a tracer. The reparation of the radioactive tracer and its paration from ^{32}P by conversion of S into FeS e described. It is shown that the major losses of in the combustion method are due to condensation SO_3 within the apparatus and to loss of SO_3 by complete absorption in the aq. absorbent. J. W. O. Pyemont

1969. **Applicability of benzoin α-oxime extraction tungsten in steel analyses. (Tests by means of dio-tungsten.)** V. Pfeifer and F. Hecht (Anal. st., Univ. Wien, Austria). *Z. anal. Chem.*, 1960, **177** (3), 175–185 (in German).—The effects of veral variables (namely, acid concn., vol., concn. W and solvents) on the extraction of W as its nzoin α-oxime complex have been investigated by eans of a tracer technique. The factors to be nsidered in the selection of the org. solvent are scussed, and in most cases the extraction of the ngsten complex is carried out three times with HCl_3. The extracted W is converted into its inol complex and determined spectrophoto- etrically at 470 mμ. The recovery of W is 93%. he interference of ions has been studied. A etailed procedure is given. B. B. Bauminger

1970. **Extraction of iron(III), cobalt and nickel ith tri-n-butyl phosphate from a hydrochloric acid olution.** Seiichiro Hikime and Hitoshi Yoshida (Chem. Dept., Fac. of Sci., Hokkaido Univ. Sapporo). *Japan Analyst*, 1959, **8** (11), 756–757.—Tervalent Fe is quant. extracted with tri-n-butyl phosphate from 2N-HCl. The separation factors for Fe/Ni, Fe/Co and Co/Ni are $3{\cdot}4 \times 10^5$ in 3N-HCl, $2{\cdot}8 \times 10^5$ in 3N-HCl, and $1{\cdot}7 \times 10^2$ in 10N-HCl, respectively. K. Saito

1971. **Note on the spectrophotometric determination of cobalt in iron and steel with nitroso-R salt.** Shigeo Wakamatsu (Toto Seiko Co., Minamisuna-machi, Koto-ku, Tokyo). *Japan Analyst*, 1959, **8** (12), 830–832.—A method of correcting the blank value is proposed. Since nitroso-R salt (**I**) does not react with Co in strongly acid soln., the reference soln. is made by boiling a second aliquot (≡ 0·05 g) of the H_2SO_4 soln. of the sample with Na acetate (50%) (20 ml, to adjust the pH to 6) and HNO_3 (sp. gr. 1·38) (10 ml) for 2 min., adding **I** soln. (2%) (10 ml), boiling for 1 min. and diluting to 100 ml. The order of addition of HNO_3 and **I** is reversed for the preparation of the test soln. The presence of <5 mg of Cu, <10 mg of V, <15 mg of Ti, <20 mg of Ni and Cr and <30 mg of Mo can then be tolerated. K. Saito

1972. **2-Thenoyltrifluoroacetone as a reagent for cobalt.** Santosh K. Majumdar and Anil K. De (Dept. of Chem., Jadavpur Univ., Calcutta, India). *Z. anal. Chem.*, 1960, **177** (2), 97–100 (in English).—Cobalt (≃25 mg) may be pptd. from aq. soln. (≃200 ml) at pH 4 to 8 by 2-thenoyltrifluoroacetone with a precision of ≃1%. The ppt. is of uncertain composition and the procedure is only advocated as a method of separation (from Ag and Hg; and from Sr, Fe^{III}, Cr^{III}, Th, Zr^{IV} and U in the presence of citrate at pH 7). Copper, Mn and Ni interfere. T. R. Andrew

1973. **Analysis of hydrocarbon solutions of cobalt carbonyl in a flowing stream.** T. F. Tsellin-skaya, N. I. Zaĭtseva and V. A. Grigor'ev (All-Union Sci. Res. Inst. of Petrothermal Processes). *Zavod. Lab.*, 1960, **26** (10), 1094–1095.—An automatic apparatus designed to measure the intensity of the colour of the soln. itself is described. It is suitable for measuring concn. of Co between 0·05 and 0·35%. G. S. Smith

1974. **Separation and determination of the platinum metals.** S. T. Payne (The Mond Nickel Co. Ltd., The Refinery, Bashley Rd., London). *Analyst*, 1960, **85**, 698–714.—After a preliminary treatment with HF, the sample is fused with Na_2O_2, the melt is leached with water, HCl is added and osmium and ruthenium are separated by distillation as the tetroxides in the apparatus described and determined absorptiometrically or gravimetrically. In the residual liquor the remaining platinum metals are complexed with nitrite, and base metals are removed by hydrolysis and ion exchange. The nitrite complexes are decomposed with $HClO_4$, and the platinum metals are separated by cellulose chromatography into fractions from which soln. are produced for determination of the metals absorptiometrically or gravimetrically. A. O. Jones

1975. **Separation of ruthenium with cation-exchange resin.** Kenjiro Hayashi, Seiichiro Hikime and Yasumitsu Uzumasa (Fac. of Sci., Hokkaido Univ., Sapporo). *Japan Analyst*, 1959, **8** (12), 821–822.—The effect of acidity upon the adsorption of Ru^{3+} and Ru^{4+} on the cation-exchange resin

Amberlite IR-120 was examined. The adsorption is increased by decrease in concn. of HCl, and $\simeq 3\%$ of Ru^{3+} is retained by the resin from 0·6N-HCl. By repeating the separation, $>99\%$ of the Ru^{3+} and Ru^{4+} can be separated from common cations.
K. Saito

1976. **Precipitation and colorimetry of ruthenium with hydrogen sulphide.** Hisateru Okuno, Hideo Yamatera and Kiyoshi Iwashima (Chem. Dept., Fac. of Sci., St. Paul Univ., Ikebukuro, Toshima-ku, Tokyo). *Japan Analyst*, 1959, **8** (11), 750–751.—The pptn. of ruthenium sulphide ($\equiv >50$ mg of Ru) is complete in 0·3 to 1N-HCl after passage of H_2S for 1 hr., or longer for higher concn. of Ru. The extinction at 372 mμ of colloidal ruthenium sulphide, prepared by passage of H_2S for 50 min. at 80° ± 2° in N-HCl, is proportional to the concn. of Ru for <3 μg per ml, in the presence of gum acacia.
K. Saito

1977. **Estimation of ruthenium species in solutions from the aqueous processing of irradiated uranium.** P. G. M. Brown and A. Naylor (U.K.A.E.A., Windscale Works, Cumberland, England). *J. Appl. Chem.*, 1960, **10** (10), 422–428.—In processed HNO_3 soln. of irradiated uranium, ruthenium is present as nitrato- (non-nitro) and nitro-complexes of nitrosylruthenium (RuNO). These can be partly separated by extraction of the sample soln. at 20° to 25° with Butex (bis-2-butoxyethyl ether) (initially equilibrated with the appropriate aq. phase), the time interval between forward extraction and first stripping being limited to 10 to 15 min. After centrifugation the phases are separated and analysed within 1 hr. for ^{106}Ru and ^{103}Ru; if mixed fission products are present, a γ-spectrometric method is used. The higher nitrato-complexes are much more readily extractable than is the mono-nitrato-dinitro-complex. The compositions of soln. of these complexes can also be determined by the paper-chromatographic procedure described previously (*cf.* Wain *et al.*, *Anal. Abstr.*, 1957, **4**, 1843), methyl isopropyl ketone (**I**) and dibutoxyethane (**II**) (each equilibrated with 3N-HNO_3) being used as eluting solvents. With **I**, $\simeq 10\%$ of the ruthenium is extracted as nitrato(RuNO) complexes and $\simeq 40\%$ as the dinitro(RuNO) complex, whilst with **II** only $\simeq 10\%$ of the Ru is extracted as the higher nitrato-complexes, the dinitro-complex being of low extractability.
W. J. Baker

1978. **Preparation and study of hydroxytriazens as analytical reagents. II. 3-Hydroxy-3-phenyl-1-*p*-tolyltriazen as a reagent for palladium and copper.** H. K. L. Gupta, T. C. Jain and N. C. Sogani (Gov. Coll., Ajmer, India). *J. Indian Chem. Soc.*, 1960, **37** (9), 531–534 (in English).—The reagent gives quant. pptn. of Cu^{2+} and Pd^{2+} between pH 2 and 3. The complexes are stable when heated, but any excess of reagent is hydrolysed by heating to yield water-soluble products that can be readily eliminated from the reaction medium. *Procedure*—To the sample soln. (120 ml) containing 10 to 20 mg of Pd or Cu add 10% Na acetate soln. (2 to 3 ml) and N-HCl (2 ml). Warm the mixture and add a 20% excess of a 1% soln. of the reagent in ethanol. Heat on a boiling-water bath for 1 hr., filter, wash the ppt. with hot water and dry to constant weight at 120° to 130°. Interference from Fe^{2+}, Fe^{3+}, Ti^{4+} and molybdate and the hydrolysis of Sn^{4+} and Zr^{4+} can be prevented by adding NaF (0·5 g), the hydrolysis of Ce^{4+} with $(NH_4)_2SO_4$, and that of Bi^{3+} and Sb^{3+} by substituting Na K tartrate f Na acetate as buffer. Results are accurate within $\simeq \pm 1\%$.
W. T. Carter

1979. **Analytical chemistry of the platinum metal VIII. Application of phosphonium and arsoniu salts to the gravimetric determination of osmiu** R. Neeb (Inst. f. anorg. Chem. und Kernchem Univ., Mainz). *Z. anal Chem.*, 1960, **177** (1), 20–3 (in German).—Solubility determinations are r ported of halogeno-osmates of substituted pho phonium and arsonium compounds, and sever are investigated for the determination of O Those recommended are tetraphenylarsoniu chloride and benzyltriphenylphosphonium chlorid in HCl soln., and tetraphenylarsonium and tetr phenylphosphonium chlorides in HBr soln.
J. H. Waton

1980. **Spectrophotometric determination of pla inum.** A. K. Majumdar and J. G. Sen Gup (Dept. of Inorg. and Anal. Chem., Jadavpur Univ Calcutta, India). *Z. anal. Chem.*, 1960, **177** (4 265–269 (in English).—Anthranilic acid forms wi Pt^{IV} at pH 5 a reddish-violet complex (1:1) havi an absorption maximum at 500 mμ. The syste obeys Beer's law for 4 to 64 p.p.m. of Pt^{IV}. Max mum colour development is attained by heatin the reactants on a steam bath for 15 to 20 mi There is no interference from Pd^{2+}, Ir^{4+}, Sr^{2} W^{6+}, Th^{4+} or Zr^{4+} present in an equal amount, from U^{6+}, Mn^{2+}, Cd^{2+} or Ba^{2+} in a 1:2 ratio, or fro Mg^{2+} or Ca^{2+} in a 1:7 ratio. EDTA (disodiu salt), Ru^{3+}, Rh^{3+}, Cr^{3+}, Hg^{2+}, Bi^{3+}, Cu^{2+}, Ni^{2} Zn^{2+}, Co^{2+} and Fe^{3+} interfere.
J. W. Price

1981. **Spectrographic analysis of blast-furna slag with a metal electrode.** Hisashi Kishitak (Chiba Plant, Kawasaki Iron & Steel Co-op Kawasaki-cho, Chiba). *Japan Analyst*, 1959, (12), 778–782.—A copper disc (diam. 40 mm thickness 5 mm) having a cavity (diam. 10 mm depth 3 mm) serves as one electrode and a syn chronous spark is struck between this and a carbo rod (diam. 6 mm). Graphite is the best buffer ar Ca is used as the internal standard. The coeff. variation is $\simeq 2\%$ for Si and Ca, 5% for Al and M and 10% for Fe, Ti and Mn. *Procedure*—Mix th sample (>200 mesh) (0·2 g) with graphite (20 mesh) (0·4 g) and starch soln. (10%) (6 drops) an dry the paste in the cavity of the disc at 11 for 1 hr. Strike a Feussner spark (primary curren 60 V, 3 amp.; secondary, inductance, 0·8 mH 0·066 μF) and expose for 60 sec. without preliminar discharge. The line-pairs used are Al 2660·3 Mg 2779·83, Mn 2933·06 and Si 2987·65 *vs.* C 2994·96; and Fe 3020·49 and Ti 3072·97 *vs.* C 3158·87.
K. Saito

1982. **Colorimetric determination of alumina iron- and steel-making slags with Solochrome cyani R [C.I. Mordant Blue 3].** P. H. Scholes and D. Smith (British Iron and Steel Res. Ass., Sheffield *J. Iron St. Inst.*, 1960, **195** (2), 190–195.—Slag suspended in boiling water and dissolved by th addition of HCl. The soln. is oxidised with HNO and evaporated to fuming with $HClO_4$ to remov fluorides. Interfering elements are removed wit NaOH - H_2O_2. The resulting soln. is neutralise and 0·5 ml of HCl (1:1) is added in excess. Th colour is developed with 0·1% aq. Solochrom cyanine R (C.I. Mordant Blue 3) in the presence H_2O_2 at pH 6·1 and measured at 535 mμ.
J. W. O. Pyemont

1983. **Complexometric analysis applied to electro-ting baths. II.** E. Julve Salvadó. *Quím. e l.*, 1960, **7** (4), 149–157.—Complexometric thods for the quantitative analysis of copper, ss, nickel and silver electroplating baths, cover- the determination of the principal metals, foreign tals and anions, are described. (52 references.) L. A. O'Neill

1984. **X-ray spectrochemical analysis: an applica-n to certain light elements in clay minerals.** W. Molloy and P. F. Kerr (Columbia Univ., w York, U.S.A.). *Amer. Min.*, 1960, **45** (9–10), 1–936.—Developments in instrumentation ex-nd the range of X-ray emission spectrographic alysis to the lighter elements, including Al, Si, Ca, P, S and Ti. Details are given for instrument libration, matrix corrections, and the use of ndards. Results are given for the application of s method to various silicate-rock specimens: e method is quant. for Ca, Ti, Cu and Pb, and mi-quant. for Si, S, K, P and Mo, but is not at esent reliable for Al. R. A. Howie

1985. **Closed-circulation systems for determining ter, carbon dioxide and total carbon in silicate cks and minerals.** P. G. Jeffery and A. D. Wilson .S.I.R., Lab. of the Gov. Chemist, Geol. Survey d Museum, London). *Analyst*, 1960, **85**, 749–755. By re-cycling air by means of a small electric mp in a closed-circuit system in the determination water, CO_2 and C in silicate rocks, the amounts of siccants can be reduced and blank values are wer than those found with conventional apparatus. the determination of water evolved at 105°, the ating chamber is maintained at 104° to 106° by eans of boiling isobutyl alcohol. In the deter-ination of total water, a gas-fired furnace at 1000° used. Alternatively, the sample is fused with a_2WO_4 and borax glass (1 : 1), the furnace temp. en being maintained at $\simeq$800°. The apparatus r the determination of CO_2 differs from that nerally used only in the inclusion of the closed-rculation system and the incorporation of a pressure amber. For total C, the method of Dixon (*Analyst*, 934, **59**, 739) is modified by the incorporation of the osed-circulation system. Results by the proposed ethods are compared with those found by con-entional procedures. A. O. Jones

1986. **Spectrometric measurement of natural and smic-ray induced radioactivity in meteorites.** . A. Van Dilla, J. R. Arnold and E. C. Anderson os Alamos Sci. Lab., Univ. California, New Mexico, .S.A.). *Geochim. et Cosmoch. Acta*, 1960, **20** (2), 5–121.—The γ-ray spectra of meteorites are easured with a large NaI(Tl) crystal scintillation ectrometer; the crystal used is 7·5 in. in diam. nd 4 in. thick. The output pulses are fed into a 0-channel pulse-height analyser. The method quires 0·5 to 1 kg or more of specimen, but is mpletely non-destructive and comparatively fast to 15 hr.). Results are given for the natural dionuclide ^{40}K and γ-emitting cosmic-ray-induced ctivation products ^{26}Al and ^{60}Co; the experimental ror appears to be $\pm$20%. R. A. Howie

1987. **Determinations of concentrations of heavy ements in meteorites by activation analysis.** W. Reed, K. Kogoshi and A. Turkevich (Argonne at. Lab., Lemont, Ill., U.S.A.). *Geochim. et osmoch. Acta*, 1960, **20** (2), 122–140.—The elements a, Tl, Pb, Bi and U and the isotopic composition certain meteorites are determined by neutron activation analysis by the techniques described earlier (*cf.* Hamaguchi *et al.*, *Anal. Abstr.*, 1958, **5**, 3723; Reed *et al.*, *U.S. Atomic Energy Comm.*, Rep. A/CONF. 15/P953, 1958). For the neutron activation determination of mercury, the radiations from the 65-hr. ^{197}Hg and 48-day ^{203}Hg formed by (n, γ) reactions on stable mercury isotopes are measured. The large neutron cross-section for the formation of ^{197}Hg more than compensates for the low isotopic abundance (0·15%) of ^{196}Hg and makes the shorter-lived activity very prominent in neutron-irradiated mercury. After the exposure to neutrons the samples are fused with Na_2O_2 in the presence of carriers: a clear melt is taken as evidence of complete equilibrium between added carriers and radioactive isotopes. R. A. Howie

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See also Abstracts—**1819**, Various applications of gas chromatography in inorg. analysis. **1820**, Applications of radioactive analysis. **1823**, Excitation of Tl and Pu. **2061**, Sulphate in biol. materials. **2063**, **2177**, ^{137}Cs, ^{89}Sr and ^{90}Sr in biol. materials and water. **2067**, Gold in biol. fluids. **2172**, Be in atmospheric dusts. **2175**, Iodide in water. **2185**, Fluoride in grass. **2207**, Grinding of rock samples. **2212**, Gas analysis. **2214**, Oxygen in gases. **2215**, Oxidising agents in gases. **2221**, Use of X-ray fluorescence for inorg. ions on paper chromatograms.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather and explosives.

1988. **The detection, identification, characterisation and determination of organic nitrogen compounds as tetraphenylborates.** A. J. Barnard, jun., and W. W. Wendlandt (J. T. Baker Chem. Co., Phillipsburg, N.J., U.S.A.). *Rev. Soc. Quim., Mexico*, 1959, **3**, 269–279.—A review of the literature covering this field, with 60 references.

1989. **Modified apparatus for the micro-determination of moisture in organic compounds.** A. C. Thomas (War Dept., Chem. Defence Exp. Dept., Porton Down, Salisbury, Wilts., England). *Analyst*, 1960, **85**, 771–772.—The apparatus is designed for removing moisture from the heated sample either by passage of dry N or by evacuation over P_2O_5. The double-ended weighing-bottle containing a platinum boat ensures a free flow of gas over the sample and is easily loaded and unloaded in a dry box. The sample is weighed before and after the determination in the weighing-bottle, so that no loss occurs by electrostatic disturbance. Electrical heating of the tube containing the weighing-bottle is effected by means of a conducting coating on the outside of the tube, the temp. (max. 350°) being controlled by a sliding resistor. A. O. Jones

1990. **Industrial analyses by the optical method. VII. Design of a non-dispersive ultra-violet photometer and its application to the examination of organic solvents.** Yoichiro Mashiko (Gov. Chem. Ind. Res. Inst., Hatagaya-honmachi, Shibuya-ku, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*,

1959, **62** (5), 640–643.—For measuring the over-all absorption of a liquid in the near u.v. region without dispersion, a simple instrument was set up. The u.v. light from a hydrogen discharge tube was passed through the sample by means of two quartz lenses and received on a photocell and the amplified current was read directly. The discharge current (300 mA) was regulated by three 25L6GT tubes to provide a stable light source. This apparatus is useful for the examination of industrial solvents (*e.g.*, benzene, acetone, methanol and CCl_4) of various grades, with the aid of suitable standards.

VIII. Application of a non-dispersive ultra-violet photometer to the quantitative analysis of binary mixtures. Yoichiro Mashiko and Hisao Konosu. *Ibid.*, 1959, **62** (5), 643–646.—By the use of this instrument, CS_2 (0·0001 to 5%) and thiophen (0·1 to 5%) in benzene are determined by means of empirical diagrams. Nitrobenzene (0·0005 to 1%) in aniline, vitamin A (1 to 30×10^4 units) in cod-liver oil and the purity of anthracene are also determined by the use of appropriate empirical diagrams. K. Saito

1991. **Continuous elemental analysis of organic compounds in gas-chromatographic effluents.** F. Cacace, R. Cipollini and G. Perez (Centro Chim. Nucl., Cons. Naz. Ric., Univ. di Roma, Italy). *Science*, 1960, **132**, 1253–1254.—A simple method for the continuous determination of the C and H content of volatile compounds, previously separated by gas chromatography, has been developed. The compounds emerging from the gas-chromatographic column are quant. converted into a mixture of CO_2 and H_2O by passage over CuO, and the H_2O is reduced to H by finely divided iron. The CO_2 and H are then separated by means of an auxiliary column and their concn. measured by a thermal conductivity cell operated at room temp. The ratio of the C to H atoms in each substance is deduced from the areas of the CO_2 and H peaks. The separation can be made and the C to H ratio determined on as little as 50 μg of organic substance in a complex mixture. R. A. Hendey

1992. **Rapid micro-analytical method for determining carbon and hydrogen in fluoro-organic compounds.** P. R. Wood (Microanalytical Lab., Pfizer Ltd., Richborough, Kent, England). *Analyst*, 1960, **85**, 764–765.—In a method developed from that of Belcher and Goulden (*Brit. Abstr. C*, 1951, 398) for the determination of C and H in fluoro-organic compounds, a NaF packing (prep. described) was used to absorb SiF_4 and was inserted in a tube connected with, but separated from, the main combustion tube. In addition to the NaF packing the tube contained silver wool and was placed in an auxiliary furnace. The normal procedure for determining C and H was then carried out, halogens and S being absorbed on the normal silver packing of the main combustion tube. The NaF was heated to 270° ± 10°. Results were accurate to within ±0·2% for both C and H. A. O. Jones

1993. **Analysis of organofluorine compounds. Simultaneous micro-determination of fluorine, carbon and hydrogen in low-boiling and gaseous compounds.** N. É. Gel'man, M. O. Korshun and K. I. Novozhilova (Inst. of Elemento-Org. Compds., Acad. of Sci., USSR, Moscow). *Zhur. Anal. Khim.*, 1960, **15** (5), 628–634.—By the use of special sampling devices the method previously published (*cf.* Korshun *et al.*, *Anal. Abstr.*, 1958, **5**, 1542) is extended to include volatile compounds. Samples of 4 to 9 mg of volatile liquid are taken in quar capillaries with an open end and for gaseous materia a gas burette is used to introduce the sample into sealed capillary. A method of opening the capillari is described. J. W. Price

1994. **Micro-determination of halogens in organ compounds.** Morizo Ishidate and Eisaku Kimur (Fac. of Pharm. Sci., Tokyo Univ., Hongo). *Japa Analyst*, 1959, **8** (11), 739–743.—Halogen evolve by the micro Carius method is absorbed in NaHSO soln. and pptd. as the silver salt, which is dissolve in $K_2Ni(CN)_4$ in an ammoniacal soln.; the displace Ni^{2+} are titrated with EDTA soln. (M/300). Th coeff. of variation is <0·25%. K. Saito

1995. **Micro-determination of halides and sulphu by the method of catalytic destructive hydrogenatio** M. N. Chumachenko and V. P. Miroshina (Sci. Re Inst. of Org. Intermediates and Dyestuffs). *Zavo Lab.*, 1960, **26** (10), 1084–1087.—The material heated in a stream of H in a quartz tube containin platinum at 850° to 900°. The H_2S and hydroge halides are absorbed in 30% KOH soln. Aliquots o the soln. are used for the various determinations b known methods. G. S. Smith

1996. **Micro-determination of sulphur in organi compounds.** Morizo Ishidate and Eisaku Kimur (Fac. of Pharm. Sci., Tokyo Univ., Hongo). *Japa Analyst*, 1959, **8** (11), 733–738.—An investigatio was made of a procedure based on combustion o the sample by the Pregl method, absorption of th SO_2 in H_2O_2, and EDTA titration in the presence o Ba. The coeff. of variation was ≃0·27%. K. Saito

1997. **Organic manganese compounds. VI. Deter mination of manganese in organic compound** R. Riemschneider and K. Petzoldt (Frei Univ Berlin-Dahlem). *Z. anal. Chem.*, 1960, **176** (6) 401–403 (in German).—*Procedure*—The compoun (200 to 250 mg) is treated with 5 to 10 ml of HNO or aqua regia in a long-necked flask heated in an ai bath until the compound is oxidised; the excess o acid is evaporated off and dil. H_2SO_4 (5 ml) con taining a few drops of 30% H_2O_2 is added; the blac ppt. completely dissolves within a few seconds The soln. of $MnSO_4$ is made up to 100 ml and a aliquot is diluted with water (400 ml). A sufficien amount of a suspension of ZnO is added to neutralis the acid and the soln. is titrated in the usual wa with 0·05N-$KMnO_4$. The error is ≯ ±0·28% (absolute). A. C. R. Hartley

1998. **Determination of higher alkoxyl groups** R. Kretz (Fa. Röhm & Haas G.m.b.H., Darmstadt Germany). *Z. anal. Chem.*, 1960, **176** (6), 421–42 (in German).—The compound (60 to 80 mg) i treated with 2 ml of 70% HI in a bomb at 120° t 130°. The solution is diluted and transferred to steam-distillation apparatus and the alkyl iodide i distilled off. Oxidation of the distillate is carrie out with Na acetate - Br - glacial acetic acid, th excess of Br is destroyed with formic acid and, afte adding KI, the liberated iodine is determined b titration with 0·1N-$Na_2S_2O_3$ in the usual way. A. C. R. Hartley

1999. **Sub-micro methods for the analysis o organic compounds. XII. Determination of ca boxyl group.** R. Belcher, L. Serrano-Berges an T. S. West (Chem. Dept., Univ., Birmingham England). *J. Chem. Soc.*, 1960, 3830–3834.—A

paratus is described in which org. acids are ated in ethanol - water (1:2) with aq. 0·01N-OH, under N. Samples (50 μg) of benzoic acid used to test the method. Direct titration with isual indicator gave a coeff. of variation of a gle result of 1·3%, back-titration with a visual icator 1·5%, direct potentiometric titration ass; calomel) 1·0%, (glass; silver - silver chloride) %, and back potentiometric titration (glass; omel) 1·2 %, (glass; silver - silver chloride) 0·8 %. e best visual indicator for a direct titration is nixture of 1 part of ethanolic 0·1% α-naphthol-halein soln. with 3 parts of ethanolic 0·1% enolphthalein soln. (pale rose to pale green at 8·6 and violet at pH 9·0). Benzoic acid, used a standard, must be dissolved as soon as it is ighed, as losses by volatilisation are consider-e. The results for several carboxylic acids are ren. E. J. H. Birch

2000. **Chemical method for the determination of rcapto-groups. Biological, toxicological and armaceutical applications.** R. Merville, J. quidt and M.-L. Corteel (Lab. de Toxicol., Fac. Méd. et de Pharm., Lille, France). *Ann. Pharm. anç.*, 1960, **18** (9), 625–633.—The determination mercapto-groups is reviewed and a modification Hellerman's method (*J. Biol. Chem.*, 1943, **147**, 3, and *Proc. Nat. Acad. Sci.*, 1953, **19**, 855) is scribed, which ensures that oxidation to disulphide ly occurs, that all mercapto-groups are freed by naturation, and that the excess of *o*-iodosobenzoic id (**I**) used as an oxidising agent does not liberate line from KI until substances capable of being idised by, or forming coloured substances with, line are removed. *Procedure*—Homogenised tissue 2 to 0·5 g) or fresh unhaemolysed serum (5 ml) in osphate buffer (4 ml) at pH 9·1 is treated with g of guanidine hydrochloride for 45 min. at room mp. The denatured material is then oxidised th a soln. of **I** (5 ml of 0·004N for tissue and 2 ml 0·002N for serum) for 5 min. under N. The protein atter is then pptd. with $CdSO_4$ and NaOH (6 ml 13·6% $CdSO_4$ soln. and 2 ml of N-NaOH for tissue d 12 ml of 27·2% $CdSO_4$ soln. and 8 ml of N-aOH for serum). The excess of **I** is then deter-ined by adding 0·1 g of KI and 5 ml of N-HCl, and rating the liberated iodine with $Na_2S_2O_3$. Pharma-utical compounds containing mercapto-groups can determined similarly, and an accuracy within 3% is attained. The method can be used for the vestigation of biologically sulphur-removing sub-ances. E. J. H. Birch

2001. **Infra-red absorption of acetylenic com-unds. Comparative study of 1- and deutero-1-kynes.** R. Romanet and B. Wojtkowiak (Lab. Chim. Générale, Fac. des Sci., Caen, France). *mpt. Rend.*, 1960, **251** (3), 364–366.—I.r. absorp-on maxima are listed to show the effect of sub-ituting ^2H for the 1-H atom in pentyne, hexyne, ptyne and octyne on the ν (CC–H), δ (CC–H) d ν (C≡C) vibrations of the hydrocarbon, either vapour or as dil. soln. in CCl_4 at 22·5°. The lculated integral absorption intensities confirm e validity of the tables compiled by Ramsay . *Amer. Chem. Soc.*, 1952, **74**, 72), and also the dependence of the CC–H vibrations in the mol. acetylenic hydrocarbons. W. J. Baker

2002. **Chromatography of epoxy-compounds. I. ne behaviour of epoxy-compounds during chro-atography and their detection on the paper.** . Schäfer, W. Nuck and H. Jahn (Inst. f. Kunst-offe, Deutsch. Akad. Wissenschaft., Berlin-Adlershof). *J. prakt. Chem.*, 1960, **11** (1–2), 1–10.—1-Chloro-2,3-epoxypropane and C_1 to C_6 alkyl glycidyl ethers are too volatile to be chromato-graphed directly, while the higher diglycidyl ethers are too hydrophobic and flow with the solvent front. Only glycidol (2,3-epoxypropan-1-ol), phenyl glycidyl ether and diglycidyl ether could be separated, and this was achieved by the ascending technique with light petroleum - n-propanol - water (1:7:2) as solvent. Whatman No. 1 or S. & S. 2043b paper was satisfactory, and the separated compounds were detected by spraying with $Na_2S_2O_3$ soln., warming at 60° to 80° for 10 min. and then spraying with bromothymol blue indicator. This is a rather insensitive reaction, and it is preferable to prepare and chromatograph derivatives of the epoxy-compounds.

II. Paper chromatography of derivatives of epoxy-compounds. W. Schäfer, W. Nuck and H. Jahn. *Ibid.*, 1960, **11** (1–2), 11–19.—The lower-mol. wt. epoxy-compounds (*e.g.*, the glycidyl ethers) may be separated by paper chromatography following their conversion into picric acid derivatives. The epoxy-compound is heated under reflux for several hours with a slight excess of picric acid and the resulting soln. is used for the test. The paper is pre-treated with 25% methanolic formamide soln., and is pressed between filter-paper to effect partial drying. The paper is then dried at 70° for 30 min. and is finally hung in the air for 60 min. A spot of soln. is applied to the paper and cyclo-hexane - toluene (2:1) is allowed to flow upwards. The compounds appear as orange spots. By exposing the paper to ammonia fumes another spot can be seen above the first; this spot fluoresces strongly in u.v. light. The two spots consist of isomers of the picric acid derivative. R_F values are quoted. H. M.

2003. **Determination of impurities in technical 1-chloro-2,3-epoxypropane by infra-red absorption spectra.** V. I. Kolbasov, S. B. Bardenshtein and R. V. Dzhagatspanyan. *Zavod. Lab.*, 1960, **26** (10), 1120–1122.—The contents of 1,2,3-trichloropropane, 2,3-dichloropropanol and 3-chloropropane-1,2-diol are determined from measurements of absorption at 15·15, 14·81 and 9·61 μ. G. S. Smith

2004. **Determination of methanol [in ethanol].** J. Kaniewski (Med. Acad., Warsaw). *Roczn. Zakl. Hig., Warsaw*, 1960, **11** (3), 241–246.—Three modifications of the method of Denigès are compared, and that of Wójcicka *et al.* (*Przem. Spożywczy*, 1957, **8**, 361) is preferred. The limit of detection is 0·03% (v/v) of methanol. The accuracy is ≃1%. *Procedure*—Place 5 ml of test soln. in a stoppered tube, add 2 ml of a 3% (w/w) soln. of $KMnO_4$ in 75% H_2SO_4, shake, and after 10 min. add 2 ml of 5% aq. tartaric acid soln. After decolorisation add 5 ml of Schiff's reagent, set aside for 1 hr. and measure the extinction with a green filter. Refer results to a calibration curve. B. K.

2005. **Contributions to fluorimetry. IV. Fluori-metric determination of glycerol after conversion into quinoline, by means of short-wave u.v. light.** J. Eisenbrand and M. Raisch (Chem. Untersuch-ungsamt f. d. Saarland, Saarbrücken). *Z. anal. Chem.*, 1960, **177** (1), 1–4 (in German).—The test soln. (1 ml, containing 0·5 to 1 mg of glycerol) is shaken with 1·1 ml of conc. H_2SO_4 and 0·25 g of reagent [aniline sulphate - Na *m*-nitrobenzene-sulphonate (4:1)] and heated at 150° to 160° with continuous stirring, for 30 min. After cooling, the soln. is transferred with 25 ml of satd. Na_2SO_4

soln. to a Parnas - Wagner nitrogen apparatus, and 10 ml of 70% KOH soln. is added. The soln. is steam-distilled into 10 ml of N-H_2SO_4 to give a vol. of 100 ml; the soln. is then diluted with 0·1N-H_2SO_4, with shaking, so that the quinoline concn. lies on the calibration curve ($0{\cdot}1 \times 10^{-6}$ to $1{\cdot}0 \times 10^{-6}$ g per ml). The fluorescence is excited by radiation of 313 mμ. Assuming a quinoline yield of 89%, the conversion factor to glycerol is 0·798. The method is satisfactory for the determination of glycerol in wine. J. H. Waton

2006. **Modification of Dische's method for the determination of tetroses.** S. Pontremoli, E. Grazi and A. de Flora (Ist. Chim. Biol. Univ., Genova, Italy). *Ital. J. Biochem.* (English Edition), 1960, **9** (4), 210–215.—To the tetrose soln. (0·5 ml) at approx. 0° add 0·1% fructose soln. (0·5 ml) and cooled H_2SO_4 - H_2O (6:1) (4·5 ml). After shaking for 3 min., set aside for 3 min. at room temp. and then place in a bath at 100° for 3 min. Cool and add a 3% soln. of cysteine hydrochloride, shake and add H_2O (1 to 2 ml). Place for 30 min. in a bath at 50° and then measure the extinctions at 458 and 480 mμ. The difference in the extinctions is a measure of the concn. of tetrose. The method was applied to D-erythrose, D-erythrose 4-phosphate and L-erythrulose. No interference was shown by D-ribose, D-fructose, D-glucose and their phosphates, nor by the products of the action of ribose 5-phosphate isomerase and phosphoketopentoepimerase on ribose 5-phosphate, nor by the products of the action of aldolase on glucose and fructose 6-phosphates. Sedoheptulose 7-phosphate and glycollaldehyde interfere. The method is valid for 0·01 to 0·06 μmole of tetrose.
J. I. M. Jones

2007. **Quantitative analysis of pentaerythritol by the X-ray method.** G. Carazzolo (Soc. Montecatini, Castellanza, Verese). *Chim. e Ind.*, 1960, **42** (8), 858–863.—The new method described is based on the examination of the sample by transmission instead of by reflection. The application of the procedure to the quant. determination of mono- and di-pentaerythritol in samples of technical pentaerythritol, with an accuracy of ±1%, is described. The method is generally applicable, particularly for low-absorbing mixtures, when the drawbacks associated with crystal orientation in the samples must be avoided. Typical X-ray diffraction curves are illustrated and discussed.
C. A. Finch

2008. **Determination of the distribution of carbon-14 by means of the decomposition of a sugar.** F. Weygand, K. Fehr and J. F. Klebe (Org.-chem. Inst., Univ., Tübingen, Germany). *Z. Naturf.*, 1959, **14b** (4), 217–220.—A ten-stage procedure is described whereby the distribution of radioactivity among the six C atoms in ^{14}C-labelled D-glucose can be determined, separately for each atom, by chemical decomposition and conversion into such compounds as AgCN (via the oxime), D-*arabino*tetrahydroxybutylquinoxaline (subsequently oxidised to its 2-carboxylic acid), 1-phenylflavazole-3-aldehyde and 1-phenylflavazole picrate. The total activity of the sample is determined by burning to CO_2 with $KClO_4$ and CuO and measuring the gas to within ±1%. The method, which is applicable to all aldo-hexoses and -pentoses, provides an easy means of control for results obtained microbiologically, and also permits determination of the distribution of activity in sugars for which no microbiological processes are available. Results for D-gluco prepared with the aid of *Escherichia coli* a *Chlorella*, respectively, are reported.
W. J. Baker

2009. **Detection and determination of 2,3-isopropylidine-L-sorbofuranose and 2,3:4,6-*O*-isopropylidene-L-sorbofuranose in mixtures paper chromatography.** M. Sterescu, S. Arizan a S. Popa (Chem. Pharm. Res. Inst., Buchares *Českosl. Farm.*, 1960, **9** (8), 398–401.—*Procedure* Submit the sample containing an 8 to 12% soln. the mixture of both compounds to chromatograp on Whatman No. 4 paper, with n-butanol - ethanc H_2O (4:1:5) as solvent system. After 4 hr. dry t paper and detect by spraying with an ethanc soln. of HCl (green - brown spots). Cut out t two spots corresponding to the mono- and compounds and extract for 15 min. with H_2O (20 m To an aliquot (1 to 5 ml) add 2N-HCl (5 ml), m and after 5 min. add 2N-NaOH (5 ml) and 42·5 NaOH soln. (2 ml), dilute with H_2O to 25 n add salicylaldehyde (0·6 ml), dilute with NaC soln. to a known vol. and measure the extinction 2,3:4,6-di-*O*-isopropylidine-L-sorbofuranose at 4 mμ. Compare with a calibration curve prepar with a known amount of acetone. Cut out anoth pair of spots, extract for 15 min. with H_2O (50 m dilute an aliquot (0·2 to 0·6 ml) to 1 ml with ethan add diphenylamine soln. (1 g in 60 ml of ethanol a 40 ml of conc. HCl) (3 ml), heat for 15 min. on water bath, then cool and dilute with ethanol 6 ml. Measure the extinction of 2,3-*O*-isopropy dene-L-sorbofuranose at 610 mμ. The method w used for the separation and determination of bo compounds as intermediates in the synthesis ascorbic acid. J. Zýka

2010. **Paper-chromatographic separation of low aliphatic aldehydes and ketones and their quantit tive determination.** G. E. Zaikov (Inst. of Che Phys., Acad. of Sci., USSR, Moscow). *Zhur. An Khim.*, 1960, **15** (5), 639–642.—Formaldehyd acetaldehyde, propionaldehyde, butyraldehyd acetone and ethyl methyl ketone are converted in the 2,4-dinitrophenylhydrazones and separat chromatographically. *Procedure*—Prepare t paper by immersing it in a soln. of toluene (1700 m acetic anhydride (300 ml) and H_2SO_4 (3 drops) 65° to 70° for 8 to 10 hr.; wash it in methanol an dry. Take $\geq 10^{-6}$ mole of the sample in methan and chromatograph for 3 to 4 hr. at 20°, wi hexane, heptane or nonane as the mobile phas Locate the spots with 5% KOH soln. in methan (**I**). Cut out the yellowish-brown spots, extra with **I** (5 ml) and measure the extinction at 480 m R_F values are given. The determinable concn. $1{\cdot}5 \times 10^{-4}$ to $17{\cdot}6 \times 10^{-4}$ mole per litre and t accuracy is within ≃10%. J. W. Price

2011. **Diazotised *p*-nitroaniline as a locati reagent for ammonium salts of aliphatic acid** A. E. Whitfield (The Hospital for Sick Childre Great Ormond Street, London, W.C.1). *J. Chr matography*, 1960, **4** (4), 350–351 (in English).- The ammonium salts of aliphatic acids can l located on paper chromatograms by spraying wi diazotised *p*-nitroaniline reagent and aq. Na_2CO The spots appear red on a pale yellow - brow background; the red colour is stable for sever weeks in a fume-free atmosphere. The reagent sufficiently sensitive for the detection of 10^{-7} mo of the ammonium salts of monobasic acids. It satisfactory with the salts of C_1 to C_9 fatty acid

2-hydroxy-4-methyl-*n*-valeric, 2-hydroxy-3-hyl-*n*-butyric α-oxoglutaric, 2-oxo-4-methyl-*n*-eric, malic, lactic, succinic, citric, tartaric, collic, tiglic, α-methylcrotonic and methacrylic ls. R. M. Rowley

012. **Polarographic determination of dissolved gen in vinyl acetate.** Shiro Usami (Toyama nt, Kurashiki Rayon Co., Toyama). *Japan alyst*, 1959, **8** (12), 807–810.—Oxygen (≃0·05 limole) in vinyl acetate (**I**) is polarographically ermined by measuring the wave height at ·5 V *vs.* the S.C.E. in 0·05M-tetramethyl-monium bromide (**II**) in a mixed solvent. nethylformamide (**III**) is the preferred solvent, apparent density being made equal to that of **I** the addition of methanol. Water of known O tent is used as internal standard. The diffusion rent is proportional to the amount of O. *Pro-ure*—Place 4·2 ml of a mixture of **III** and thanol (3:2) and 2·45 ml of aq. 0·15M-**II** in a cell l measure the current at −0·5 V in an atmosphere N. Add **I** (0·35 ml) and again measure the current. culate the concn. of O from the difference.

K. Saito

013. **Limit of determination of minor constituents raw materials for the production of poly(vinyl ohol) by various spectrophotometric methods.** taka Nishino (Toyama Plant, Kurashiki Rayon , Toyama). *J. Chem. Soc. Japan, Ind. Chem. t.*, 1959, **62** (5), 662–666.—Data are given for the ermination of methanol, methyl acetate, vinyl tate, acetic acid, isopropyl acetate, acetaldehyde, tone, crotonaldehyde, benzene, toluene and inylacetylene by u.v. and i.r. spectrophotometry l mass spectrometry. K. Saito

014. **The hydrolysis and analytical control of ric acid esters.** E. Schulek, K. Burger and M. nér (Inst. Inorg. and Anal. Chem., L. Eötvös iv., Budapest). *Z. anal. Chem.*, 1960, **177** (2), -86 (in German).—Glycerol trinitrate, penta-thritol tetranitrate, starch nitrate and cellulose rate have been hydrolysed in 0·4N-ethanolic OH and the hydrolysates examined for CN^-, H_4^+, NO_3^- and NO_2^- by previously published thods (*Anal. Abstr.*, 1960, **7**, 451). The relative ounts of the several N-containing compounds sent serve to distinguish the four compounds etone is used for the hydrolysis of cellulose rate). Pentaerythritol tetranitrate is unique in ing no CN^- on hydrolysis. T. R. Andrew

015. **Cerimetry in non-aqueous media.** G. abhakar Rao and A. R. Vasudeva Murthy st. of Sci., Bangalore, India). *Z. anal. Chem.*, 60, **177** (2), 86–89 (in English).—Potassium ethyl-nthate (5 to 25 mg) in methyl cyanide may be rated with ceric ammonium nitrate (≃0·05N) methyl cyanide, with a precision of about 1%, e orange colour of the Ce^{IV} soln. serving as licator. For larger amounts of xanthate (75 150 mg) the error increases to about 10%.

T. R. Andrew

016. **Determination of phenyl radicals in organo-icon compounds.** A. P. Kreshkov, V. T. Shem-tenkova, S. V. Syavtsillo and N. A. Palamarchuk . I. Mendeleev Moscow Chem-Tech. Inst.). *ur. Anal. Khim.*, 1960, **15** (5), 635–638.—*ocedure*—To 2 to 2·5 g of the compound add, per enyl radical present, 6 to 7 g of anhyd. $AlCl_3$ d 35 to 40 g of ethyl bromide and maintain under flux for 2 hr. at 30°. Add 50 ml of H_2O, cool and extract the hexaethylbenzene with ether (5 × 50 ml). Wash the extract with H_2O and distil off the solvents. Dry over P_2O_5 and weigh. Calculate the phenyl radical content by using an empirical coeff. of ethylation of 0·91. J. W. Price

2017. **Analysis of organosilicon compounds containing nitrogen and carboxyl by titration in non-aqueous media.** A. P. Kreshkov, V. A. Drozdov and E. G. Vlasova (D. I. Mendeleev Moscow Chem.-Tech. Inst.). *Zavod. Lab.*, 1960, **26** (10), 1080–1084.—With N-containing compounds, the sample (0·04 to 0·1 g) is dissolved in 6 ml of methyl cyanide or nitromethane, an equal vol. of benzene or dioxan is added, and the soln. is titrated potentiometrically with 0·1N-$HClO_4$ in glacial acetic acid. Alternatively, the end-point may be found visually by use of one of a number of suitable indicators, *e.g.*, crystal violet or bromocresol purple. With carboxyl-containing compounds, the soln. in the mixture of organic solvents is titrated with a soln. of tetraethylammonium hydroxide in benzene - methanol. G. S. Smith

2018. **Determination of chlorine in tetrabutyl titanate.** Ya. I. Chulkov. U.S.S.R. Pat. 128,190 (28th April, 1960).—The method is based on conversion of Ti into TiF_6^{2-} and determination of Cl by known methods. Dissolve the sample (0·8 to 1·0 g) by stirring and heating to between 110° and 140° with excess of a mixture of 15 ml of acetic anhydride and at least 4 g of NH_4F (>2 g of NH_4F per 100 ml of soln.), dissolve the ppt. in the minimum of water, heat the mixture to between 50° and 55° with stirring, cool and make up to 250 ml. Use this soln. for the determination of Cl by titration with $AgNO_3$ or by adding excess of $AgNO_3$ and titrating the excess with NH_4SCN; details of the titrations are given. C. D. Kopkin

2019. **Mass-spectrometric analysis of ring-deuterated pentamethylbenzene.** N. Martalogu, D. Mumuianu, M. Magda and A. T. Balaban (Inst. Atomic Phys., Acad. R.P.R., Bucharest). *Acad. R.P.R., Stud. Cercet. Chim.*, 1960, **8** (2), 329–338.—The ^2H content of ring-labelled pentamethylbenzene (**I**) used in kinetic measurements was determined with a MI-1305 mass spectrometer. Samples of isotopically labelled and unlabelled **I** were analysed, with $ZnCl_2$ as standard for mass numbers, at an ionising tension $V_i = 20$ V. Authors' Summary

2020. **Diphenylpicrylhydrazyl as a reagent for phenols in paper chromatography.** H. Herzmann and P. Venker (Dtsch. Akad. d. Wissenschaften, Berlin). *Z. Chem.*, 1960, **1** (1), 29.—A 5×10^{-4}M soln. of 2,2-diphenyl-1-picrylhydrazyl in acetone is a useful spray reagent for detecting microgram amounts of phenols on paper chromatograms. The phenols show up as violet flecks. There is no reaction with *m*-hydroxyphenols, *e.g.*, resorcinol, phloroglucinol, or with monohydric phenols. The reaction thus provides a useful method for differentiating phenols of various types. S. M. Marsh

2021. **Colorimetric determination of phenols with *p*-nitrobenzenediazonium fluoroborate.** E. Leibnitz, U. Behrens, L. Riedel and A. Gabert (Inst. f. Verfahrenstechn. d. org. Chem., Leipzig, Germany). *J. prakt. Chem.*, 1960, **11** (3–4), 125–132.—The method is used for the determination of mixtures of *m*- and *p*-cresols, which are said to be inseparable by paper chromatography. The sample soln. is divided into two parts, one of which is adjusted to

pH 12, the other being adjusted to pH 9·5. To each are added 1 ml of 0·2% reagent soln. (prep. described) and 5 ml of 1% dextran soln. and each is diluted to 100 ml. The extinctions are measured, that of the soln. at pH 12 being due to *m*-cresol alone, while that of the soln. at pH 9·5 is due to both *m*- and *p*-cresols. Measurements made with pure *m*-cresol at both pH values enable the extinction at pH 9·5 to be corrected for the *m*-cresol present. The *p*-cresol content can then be calculated. H. M.

2022. **Quantitative column-chromatographic determination of catechols in technical products.** E. Leibnitz, U. Behrens and H. Czech (Inst. f. Verfahrenstechnik d. org. Chem., Leipzig). *J. prakt. Chem.*, 1960, **11** (1–2), 73–81.—Catechol can be separated from its homologues and isomers in phenolic oils by column chromatography on cellulose powder. The preparation of a suitable grade of powder is described. The column (150 cm × 1 cm) is filled to a height of 120 cm with the powder formed into a slurry with the mobile phase (the lower layer of a mixture of 97·5 parts of CCl_4 and 2·5 parts of n-butanol with 100 parts of H_2O). The sample (> 5 ml) dissolved in the mobile phase and containing ≃ 30 mg of catechol is run on to the column and eluted, the eluate being collected in 3-ml portions. To each portion are added 2 ml of reagent (0·45 g of $FeSO_4.7H_2O$ and 1·25 g of K Na tartrate dissolved in 250 ml of H_2O), 10 ml of H_2O and 10 ml of buffer (24 g of $Na_2HPO_4.12H_2O$ dissolved in 1 litre of H_2O). The soln. is shaken and the lower layer is separated and the extinction measured. The catechol appears between fractions 110 and 145. A smaller column (30 cm high) will suffice if only catechol is to be determined. H. M.

2023. **Determination of mono- and di-potassium *p*-hydroxybenzoate by potentiometric titration.** Ryuzo Ueno (Ueno Pharm. Ind., Komabashi, Higashi-ku, Osaka). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1960, **63** (2), 287–288.—Dipotassium *p*-hydroxybenzoate is potentiometrically titrated with HCl soln., the equilibrium points being at pH 7 and 3; K salicylate interferes. Monopotassium *p*-hydroxybenzoate is similarly titrated with KOH soln. (the equilibrium point being at pH 11·5); K salicylate does not interfere. The method is applicable to the analysis of the product of the Kolbe - Schmitt reaction. K. Saito

2024. **Quantitative determination of *cis*- and *trans*-but-2-ene-1,4-diol dibenzoates.** R. Riemschneider, G. Pauling and W. Ernst (Freie Univ., Berlin-Dahlem). *Z. anal. Chem.*, 1960, **177** (1), 35–36 (in German).—The proportions of these geometric isomers are determined from the measurement of the extinction of a soln. of the material in CS_2 at 7·45 μ. J. H. Waton

2025. **Polarography of 5-*p*-nitrophenyl- and 5-*p*-aminophenyl-azosalicylic acids.** É. S. Levin and Z. I. Fodiman (Sci. Res. Inst. of Org. Intermediates and Dyestuffs). *Zavod. Lab.*, 1960, **26** (10), 1090–1093.—In an acetate buffer soln. at pH 4·3, 5-*p*-aminophenylazosalicylic acid (**I**) gives a polarographic wave with $E_{\frac{1}{2}} = -0{\cdot}32$ V *vs.* the S.C.E. that is due to reduction of the azo-group, and 5-*p*-nitrophenylazosalicylic acid (**II**) gives a wave at −0·17 V due to the azo-group and another at −0·54 V due to the nitro-group. At pH 7·2 the corresponding $E_{\frac{1}{2}}$ values are −0·56 V, −0·28 V and −0·68 V, and at pH 9·2 the values are −0·83 V, −0·44 V and −0·93 V. With mixtures of **I** and at pH 4·3 the azo-group waves form one wa whilst the nitro-group wave is distinct, at pH the three waves are superimposed, and at pH the wave due to the azo-group of **II** and the co bined wave due to the azo-group of **I** and the nit group of **II** are clearly defined and suitable measurement. The number of electrons concern in the reductions are 2 for the azo-group and 6 for t nitro-group. G. S. Smith

2026. **Test for the detection of aromatic amin** W. E. Hearn and R. Kinghorn (Chem. Inspec Min. of Aviation, H.Q. Building, Royal Arsen Woolwich, London). *Analyst*, 1960, **85**, 766–768. Ceric salts can produce highly coloured oxidati products with aromatic amines dissolved in a varie of organic solvents. The reproducible developme of the colour is dependent on the relative amour of solvent, ceric reagent and amine present. T amine (0·05 g of the free base or its approx. equi of salt) is dissolved in 100 ml of acetone - wat (3 : 1), and to 10 ml of this soln. are added two dro only of a soln. of 10 g of ammonium ceric nitrate 100 ml of 5% HNO_3. The colour formed is not before and after warming the mixture for 30 60 sec. The colours produced with 33 amines a tabulated. Aliphatic amines and other N-containi compounds give no colour. The test may ha value in distinguishing isomers of aromatic amine A. O. Jones

2027. **Determination of anthracene. III. Erro in the chlorobenzene partition method.** Eiic Funakubo (Chem. Inst., Tech. Fac., Univ. of Osak Japan). *BrennstChemie*, 1960, **41** (8), 243–246. Sources of error are considered and their cumulati effect on the accuracy limits are expressed equations and graphs. Since the accuracy depen on the anthracene content of the sample, a pr liminary test is recommended. A. R. Pearson

2028. **Polarographic control in the catalyt manufacture of anthraquinone.** Z. I. Fodiman a É. S. Levin (Sci. Res. Inst. of Org. Intermediat and Dyestuffs). *Zavod. Lab.*, 1960, **26** (10), 108 1090.—To determine anthraquinone, the sample dissolved in H_2SO_4 and an aliquot is diluted wi water to 12 ml and then with ethanol to 100 ml that the final acid concn. is 0·7N. After removal O, the polarographic wave at $E_{\frac{1}{2}} = -0{\cdot}5$ V recorded. Alternatively, the material is dissolved 100 ml of a soln. prepared by dissolving 5·35 g NaCl in 100 ml of water and diluting the soln. 1 litre with pyridine. The polarographic wave $E_{\frac{1}{2}} = -0{\cdot}25$ V is measured. To determine anthr cene, 10 ml of a benzene soln. is mixed with 3 ml an aq. soln. containing 10·7 g of tetraethylammoniu iodide in 50 ml and then diluted to 50 ml wi ethanol. The wave at $E_{\frac{1}{2}} = -1{\cdot}9$ V is measured. G. S. Smith

2029. **Determination of 5-ethyl-2-methylpyridi in commercial 2-methyl-5-vinylpyridine.** Yu Takayama (Mitsubishi Rayon Co., Kyobash Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sec* 1959, **62** (5), 658–661.—5-Ethyl-2-methylpyridi (**I**) (3 to 15%) in 2-methyl-5-vinylpyridine (**II**) determined by u.v. spectrophotometry in 3% a H_3PO_4 soln. by the use of the empirical formula $D_1 = 0{\cdot}001797x + 0{\cdot}005397y$, and $D_2 = 0{\cdot}003748$ where D_1 and D_2 are the extinction coeff. at 270 a 292·5 mμ, respectively, and x and y are the quantiti of **II** and **I** (μg per 10 ml), respectively. **I** (> 0·01%

II is determined by gas chromatography at 9° with Carbowax 400 as stationary phase and e (60 ml per min.) as carrier gas. **I** and **II** are so separated by column chromatography, with ght petroleum as developing agent and alumina ntaining $CuCl_2$ as adsorbent. Commercial acti- ated alumina is boiled with 2N-HCl for 2 hr., ashed and dried at 400° to 500° for 3 hr.; a 125-g ortion is mixed with a mixture (50 g) of aq. $CuCl_2$ ln. (10 g per 10 ml) and glycerol (2:3, w/w) and ried at 50° for 5 hr. K. Saito

2030. **Benzotriazole (photographic grade).** British tandards Institution (2 Park Street, London, W.1). .S. 3309:1960. 7 pp.—Tests for purity and a ethod of assay are given.

2031. **Determination of water in gaseous hydro- rbons: Fischer reagent method.** Petroleum Gases anel, Institute of Petroleum (61 New Cavendish treet, London, W.1). *J. Inst. Petrol.*, 1960, **46**, 94–298.—The sample is passed through the tration vessel, fitted with two sealed-in platinum ectrodes and containing ethanediol and an excess Karl Fischer reagent; the end-point is deter- ined by the dead-stop method. The apparatus, ethod of operation, and circuit of the titrimeter re described. The method, suitable for hydrocar- on gases and some liquefied petroleum gases, is pplicable to water concn. in the range of a few p.m. to 2 mg per 100 ml. D. DeVoy

2032. **Complete analysis of gaseous hydrocarbons y gas adsorption - partition chromatography.** obuyoshi Hara, Hiroshi Shimada, Akira Ishikawa nd Kunio Dohi (Tokyo Inst. of Technol., Meguro, okyo). *Bull. Japan Petrol. Inst.*, 1960, **2**, 33–40 n English).—A 15-component mixture of gaseous ydrocarbons can be completely analysed in 30 min. n a 4-metre column containing dimethylformamide 0%) and squalane (1·5%) on alumina ignited r 2 hr. at 700°. The column is used at room temp. ith H or He as carrier gas. The dimethylform- mide does not evaporate from calcined alumina. G. Burger

2033. **Mass-spectrometric analysis of hydrocarbons eparated by gas chromatography.** Shozo Oshima, kira Katsumata and Toyosaburo Dan (Central es. Lab., Maruzen Sekiyu Co., Shimotsu, Waka- ama-ken). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 959, **62** (5), 646–649.—Various compounds eparated by gas chromatography (with carrier elite 408, 35 to 60 mesh; stationary phase, squalane; arrier gas, H; rate of flow, 65 ml per min.; length f column, 15 metres; diameter, 5 mm) are collected y a liquid-nitrogen trap and submitted to mass pectrometry at a pressure of 0·001 to 0·1 torr. The ydrocarbons are classified into four groups (aro- natics, normal paraffins, isoparaffins and naphth- nes); the number of carbon atoms is determined and ach isomer is identified in the usual way. This ethod is useful for the analysis of paraffins and aphthenes in the 46° to 80° fraction in platformate, nd of total aromatic compounds in all platformates. K. Saito

2034. **Gas-chromatographic determination of romatic compounds in platformate.** Toshihide Iaeda and Mitsuharu Fujii (Iwakuni Plant, Mitsui etrochem. Ind., Waki-mura, Kuka-gun, Yama- uchi-ken). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 959, **62** (5), 649–652.—Gas chromatography of benzene, toluene, ethylbenzene, and *o*- and *p*-xylenes in the presence of raffinate was studied by the use of the Perkin-Elmer fractometer (stationary phase, kieselguhr impregnated with an addition compound of picric acid and fluorene; carrier gas, He; rate of flow 86 to 89 ml per min.; temp., 100°). The standard deviations of the result are, respectively, 0·5, 0·2, 0·3, 0·3 and 0·7% for 20% of each of the named compounds in the order given. This method is applicable to the analysis of platformate. K. Saito

2035. **Complexometric determination of tetra- ethyl-lead in petrol.** É. I. Uvarova and N. M. Vanyarkina. *Zavod. Lab.*, 1960, **26** (10), 1097–1098. —The sample (10 to 50 ml) is treated with a 30% soln. of Br in CCl_4 until the brown colour persists for 5 min. The ppt. is dissolved and a homogeneous soln. is obtained by addition of methanol. The liquid is boiled and N-ethanolic KOH is added until the brown colour is discharged and then 3 to 4 ml more. After addition of 200 ml of water the two- phase system is boiled under reflux. If the soln. becomes acid to methyl red a further amount of ethanolic KOH soln. is added and the boiling continued. After this, an excess of 0·03N-EDTA (disodium salt) is added, the soln. is neutralised with dil. H_2SO_4, 10 ml of a buffer soln. (350 ml of 25% aq. NH_3 and 54 g of NH_4Cl dissolved in 1 litre) and Eriochrome black T - NaCl (1:99) indicator are added, and the soln. is titrated with 0·03N- $ZnSO_4$ to a colour change from greenish blue to violet. G. S. Smith

2036. **Spectrographic determination of vanadium and nickel in petroleum products.** K. M. Vaĭsberg and V. G. Zizin (Bashkiria Sci. Res. Inst. of Petrol- eum Refining). *Zavod. Lab.*, 1960, **26** (10), 1123–1124.—The material is ashed and the residue is dissolved in HCl. The acid is evaporated off and the salts are dissolved in a suitable volume of water. The soln. is placed in a special reservoir from which it flows up a hole in a carbon electrode and then over the upper surface. The spectrum is excited by means of a spark between this electrode and an upper carbon electrode. Sample and standard soln. are mixed with a soln. containing Ti, which serves as internal standard for the determination of V and Ni. G. S. Smith

2037. **Elementary analysis and calorific value [of fuels].** A. Härlin (Swedish Royal Acad. of Engng Sci., Stockholm). *Acta Polytech. Scand., Phys. Nucleonics Ser.*, 1960, **3** (5), 3–14 (in German).— A survey is given of various proposed formulae relating the composition of a fuel to its calorific value. A number of simple formulae take the form

$$K_b = c.\mathrm{C} + h.\mathrm{H} - o.\mathrm{O}$$

where K_b is the calorific value in kcal. per kg, C, H and O are the percentages of these elements in the fuel, and c, h and o are constants. Different values for these exponents, especially for o, are given by different authors. More complex formulae have also been proposed, in which the values of the factors depend on the analysis. Data from about 600 fuels are considered. Groups of these with similar C contents have their H contents plotted against calorific value. It is concluded that the simple equation giving the best fit is $K_b = 82\mathrm{C} + 280\mathrm{H} - 29\mathrm{O} + 200$. The value of (82C + 280H) should not exceed 7900. Calculated results agree with those determined to within a few per cent. G. Russell

2038. **Rapid determination of sulphur in fuel oils used in the steel industry.** W. B. Boyes and H. C. Wilkinson (Brit. Coke Res. Ass., Coke Res. Centre, Chesterfield). *Metallurgia, Manchr*, 1960, **62**, 129–130.—The calorimeter-bomb method and the Sheffield high-temp. method were studied. Combustion in oxygen in a calorimeter bomb, followed by titrimetric determination of the sulphur, gave good results. The Sheffield high-temp. method, extensively used to determine sulphur in coke, can be adapted for use with fuel oils by alteration of the combustion time and oxygen flow-rate. Results from the two methods are in fairly good agreement, the bomb method being more accurate.

M. G. SEAMAN

2039. **Qualitative and quantitative determination of nitromethane in fuel for small Diesel motors.** A. Blumenthal (Chem. Lab. der Stadt, Zürich, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1960, **51** (5), 359–365 (in German).—*Procedure* (*quant.*)—Weigh 0·5 g of the fuel, add 10 ml of light petroleum and 25 ml of water, stir magnetically for 15 min., add water until the aq. phase is 500 ml and discard the org. phase. Add 2 ml of 2% aq. NH_3 and 5 ml of 2% methanolic vanillin soln. to a graduated 20-ml test-tube, then 1 to 3 ml of the aq. phase, shake and set aside in a water bath at 60° for 30 min. After cooling make up to 20 ml with water and measure the extinction at 500 mμ. A standard curve is given. *Procedure* (*qual.*)—Add 2 ml of 2% aq. NH_3, 5 ml of vanillin soln. and 0·1 ml of fuel to a graduated test-tube and proceed as described above. Nitromethane is present if the aq. methanolic phase has a reddish-violet colour; 0·1% of nitromethane can be detected.

I. DICKINSON

2040. **Analysis of petroleum hydrocarbons by mass spectra.** Toyosaburo Dan and Shozo Oshima (Maruzen Oil Co., Shimotsu, Wakayama, Japan). *Bull. Japan Petrol. Inst.*, 1960, **2**, 25–32 (in English).—A scheme is described for the identification by mass spectrography of individual hydrocarbons after their separation by gas chromatography. From the tabulated data, it is possible first to determine the hydrocarbon type, then the carbon number and finally to identify the isomer.

G. BURGER

2041. **General method for the chromatographic analysis of mono-, di- and tri-glycerides and the mono- and di-esters of ethanediol and polyoxyethylene glycol.** G. J. Papariello, S. Chulkaratana, T. Higuchi, J. E. Martin and V. P. Kuceski (Univ. of Wisconsin, Madison). *J. Amer. Oil Chem. Soc.*, 1960, **37** (8), 396–399.—Esters of glycerol, ethanediol and polyoxyethylene glycol have been separated on a silica-gel column by elution with a series of increasingly polar solvent mixtures. With commercially available products, a nearly quant. recovery was achieved in most cases. Small amounts of monoglycerides (approx. 1%) were determined separately after extracting the mixture with an equal quantity of abs. ethanol. Esters of a given acid with polyoxyethylene glycols of different mol. wt. (e.g., 400 and 600) do not appear to give separate peaks.

P. M. KINGSTON

2042. **Adaptation of the chromotropic acid method to the assay of Spans.** L. Gatewood, jun., and H. D. Graham (George Washington Carver Found., Tuskegee Inst., Ala., U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (10), 678–680.—The method depends on saponification of the particular Spa (sorbitan monolaurate, palmitate or stearate) yield sorbitol, which is then oxidised by periodat to formaldehyde; this gives a wine-red colour wh made to react with chromotropic acid. The r agents should be prepared daily according to t method of West and Rapoport (*Proc. Soc. Ex Biol. Med.*, 1949, **70**, 141). Satisfactory recovery obtained in the presence of anionic but not cation detergents. *Procedure*—Heat the sample (1 g) a boiling-water bath for 20 min. with 0·5M-KO (50 ml), cool and dilute with H_2O. Mix a 1-r aliquot containing the equivalent of $\simeq$0·4 mg the original Span with 0·0075M-periodic acid reage (0·5 ml), set aside at 28° $\pm$ 1° for 8 min., ad 0·0035M-$SnCl_2$ reagent, mix again and cool. Ad chromotropic acid reagent (6 ml) and H_2O (2 ml heat in boiling water for 30 min., cool and measu the extinction at 570 mμ. Perform a reagent blan

A. R. ROGERS

2043. **Estimation of free alcohols in essential oil** P. S. Ramanathan (Ess. Oils Grading Schem Cochin). *J. Instn Chem., India*, 1960, **32** (3), 130–13 —The accuracy of the standard method (IS 326—1952) for calculating the percentage of fre alcohols in oils containing free acid is disputed, an a new formula is proposed, *viz*, free alcohols (% (as a monohydric alcohol of mol. wt. M) = $M(S - S_1)/0{\cdot}42(1336 - S)$, where S_1 and S are th sap. values before and after acetylation, respectively It is recommended that the NaCl soln. used fo washing the acetylated oil should not contai Na_2CO_3. The application to the analysis of vetive oil is described.

P. M. KINGSTON

2044. **Determination of eucalyptol [cineole] b residual titration with hydrogen bromide in aceti acid.** M. I. Blake and G. Rabjohn (School of Pharm N. Dakota Agric. Coll., Fargo, U.S.A.). *J. Amer Pharm. Ass., Sci. Ed.*, 1960, **49** (10), 650–651.— Dissolve the sample (100 to 200 mg) in glacia acetic acid (20 ml), add 0·1N-HBr in acetic aci (25 ml), stopper the flask tightly, set aside for 48 h at room temp. and titrate rapidly with 0·1N-N acetate in acetic acid, with methyl violet as ind cator. Perform a blank titration with each serie of 4 to 6 samples. The recovery in 8 determination was 99·86 $\pm$ 0·11%. No reaction is observed wit camphor, menthol or thymol. The method cannc be used for the determination of cineole in eucalyptu oil, because other unsaturated constituents react t give high results.

A. R. ROGERS

2045. **Quantitative determination of azo dyes b direct reductimetric titration with vanadium(II sulphate.** M. Matrka and Z. Ságner (Res. Inst Org. Synth., Pardubice-Rybitví, Czechoslovakia *Chem. Prùmysl*, 1960, **10** (9), 474–475.—Th accuracy of the direct reductimetric determinatio of azo dyes with VSO_4 is limited by the fact that th azo-group reacts not only with the volumetri reagent, but also with V^{III} formed during th reaction, so that a visual determination is nc satisfactory. The course of the suitable potentio metric titration is explained and illustrated by th titration of methyl red in K Na tartrate mediur at 90° in a nitrogen atmosphere.

J. ZÝKA

2046. **Paper-chromatographic analysis of inks dyestuffs and lipsticks and their importance i crime-detection.** S. N. Tewari [18/3A, The Mal Kanpur (U.P.) India]. *Z. anal. Chem.*, 1960, **17**

408–412 (in English).—Twelve types of foun- n and ball-pen inks were extracted from docu- nts with acid ethanol. Spots (0·001 ml) of the racted and control inks were used for chromato- phy on Whatman No. 1 paper by the ascend- technique with n-butanol - acetic acid - water 1:5) as solvent. After drying, the chromato- ms were examined in daylight and u.v. light. ie lipsticks were examined. The dyes were arated from the fatty matter by extraction with it petroleum from acetic acid soln., dissolved in % ethanol and chromatographed. R_F values and ours are tabulated. A. C. R. HARTLEY

2047. **Determination of a small amount of ucing substance in cellulose with semicarbazide.** nzaburo Yamamoto and Toru Miyata (Res. st., Tohoku Pulp Ind., Araya-cho, Akita). *J. em. Soc. Japan, Ind. Chem. Sect.*, 1959, **62** (9), 33–1435.—For the application of the semi- rbazide (**I**) method to micro-analysis, the reagent n. is best buffered with phthalate. The decom- sition of **I** is more rapid at a high pH value, the timum for analysis being 4·0. *Procedure*— gest the sample (0·6 g) in **I** soln. (50 ml) for 48 hr., er and wash the pulp with water (150 ml). To 50-ml portion add 6N-H_2SO_4 (30 ml) and 0·04N- IO_3 (25 ml), shake for 10 min., add KI soln. %) (10 ml) and titrate with 0·02N-$Na_2S_2O_3$. etermine the blank similarly. For the preparation **I** soln., dissolve **I** hydrochloride (1·740 g) in 2N-K H phthalate (250 ml), adjust the pH to 4·0 th NaOH and dilute to 1 litre. K. SAITO

2048. **Tentative textile standard number 63. uantitative chemical analysis of binary mixtures of rylic and certain other fibres.** Textile Inst. (10 ackfriars Street, Manchester, England). *J. Text. st. Proc.*, 1960, **51** (9), P566–P570.—The method applicable to binary mixtures of acrylic fibres th wool, silk, cotton, polyamide, polyester and scose rayon fibres, in any textile form, *e.g.*, tow, ver, roving, yarn or fabric. It is not applicable acrylic fibres dyed with pre-metallised dyes. ie acrylic fibres are removed from a known y-weight of the mixture by dissolving in di- ethylformamide at 60°. The residue is collected, ashed, dried and weighed; its weight is corrected, nen necessary, for loss in the pre-treatment, and pressed as a percentage of the total dry-weight fibre in the mixture. The proportion of acrylic re is found by difference. O. M. WHITTON

2049. **New method for detecting the presence and pth of penetration of boron in treated timber.** Cockcroft (Forest Products Res. Lab., Princes sborough, England). *Holzforschung*, 1960, **14** , 117–119 (in English).—An improved method is scribed, in which the qual. test of Wirth (*Anal. bstr.*, 1955, **2**, 1776) is adapted for use as a spot st. Two reagent soln. are required, *viz*, (*i*) a % aq. soln. of poly(vinyl alcohol) acidified with equal volume of 10% HCl, and (*ii*) 0·1N-iodine. ie soln. are sprayed consecutively on to a cross- ction of the test sample. A dark-blue colour is tained where the wood has been penetrated by e boron preservative. The test is equally satis- ctory for freshly treated or dry timber. Limits of nsitivity on freshly treated and dried timber are 15 lb per cu. ft. and $\simeq$0·04 lb per cu. ft., respec- vely, as boric acid. The test is claimed to be as mple as, but more rapid than, the conventional rmeric test. S. D. ROSS

2050. **Analysis and testing of plastics.** G. C. Ives. *Fibres and Plastics*, 1960, **21** (10), 307–310, 313.—A review with 32 references.

2051. **Analysis of plasticisers by micro-adsorption chromatography.** J. W. Copius Peereboom (Gov. Dairy Sta. Leiden, The Netherlands). *J. Chromatography*, 1960, **4** (4), 323–328 (in English).—A number of plasticisers, including several that have been approved for use in food-packaging materials, can be separated by chromatography on a mixture of silica gel and plaster of Paris, the technique described by Stahl (*ChemikerZtg*, 1958, **82**, 323) being used. The solvents used are iso- octane with 10% of ethyl acetate, benzene with 5% of ethyl acetate, and dibutyl ether with 20% of hexane. Several pairs of plasticisers cannot be separated and must be identified by colour tests, a number of which are given. Plasticisers based on natural glycerides give a series of spots, and interfere with the identification of other plasticisers.
R. M. ROWLEY

2052. **Determination in vinyl esters of mercury derived from mercuric acetate catalyst.** C. E. R. Jones and G. E. J. Reynolds (Vinyl Products Ltd., Carshalton, Surrey, England). *Analyst*, 1960, **85**, 768–769.—To 15 ml of nitrobenzene in a 3-necked round-bottomed litre flask, equipped with stirrer, condenser and gas bubbler, are added 250 g of the sample (vinyl acetate or soln. of higher esters), 6 ml of glacial acetic acid and 9 ml of 2N-HCl and H_2S is passed through with rapid stirring for 45 min., *i.e.*, 15 min. through the cold liquid to fix organic mercurials, 15 min. while the temp. is being raised to b.p. on a water bath, and finally through the liquid boiling under reflux. The H_2S flow is then stopped, boiling ethanol (250 ml containing 10 ml of nitrobenzene) is added and stirring is continued for 5 min. The ppt. is collected in a sintered glass crucible with suction, washed under gravity successively with boiling ethanol, CS_2 and ether, dried at 110° to 120°, and weighed. A. O. JONES

2053. **Analysis of urea - formaldehyde resins by means of column chromatography and infra-red absorption spectrophotometry.** Shigeyuki Tanaka, Yasuo Miyamoto, Naoaki Yoshimi and Yasuaki Matsuda (Inst. Tech.-anal. Chem., Fac. of Engng, Tokyo Univ., Hongo, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1959, **62** (5), 653–658.—Water-soluble condensation products of urea and formaldehyde at the earlier stage are fractionated into two parts by column chromatography with filter-pulp powder as the column substance, a mixture of ethanol (**I**), methanol (**II**) and water (3:1:1) as developing agent, and a mixture of **I**, **II** and water (3:1:16) as eluting agent. The first fraction consists of mono- and di-(hydroxy- methyl)urea; both are determined by i.r. spectro- photometry by the potassium bromide pellet method (Tanaka *et al.*, *Anal. Abstr.*, 1958, **5**, 2721), the standard deviation being 1·7%. The second fraction contains various condensation products other than methylenediurea and di(hydroxymethyl)- methylenediurea. K. SAITO

2054. **Analysis of silicones by optical methods. I. Analysis by ultra-violet absorption spectrophoto- metry.** Toshizo Uriu and Tomomi Hakamada (Central Res. Lab., Shin-etsu Chem. Ind., Iriarai, Ota-ku, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1959, **62** (9), 1421–1428.—While siloxanes containing only methyl groups as org. components have no absorption in the region $>$240 mμ, those

containing phenyl groups adjacent to Si have marked absorption at 270, 264, 259 and 253 mμ. The molar extinction coeff. corresponding to one phenyl group is practically constant (335) at 264 mμ for high-mol.-wt. siloxanes. This is useful for the determination of the phenyl group. Compounds in which two phenyl groups are attached to Si have much greater extinction at <240 mμ than those in which one phenyl group is attached to Si. These findings can be applied to the analysis of silicone lubricants, silicone varnish and silicone rubber.

II. Analysis of methylsilicones by infra-red absorption spectrophotometry. Toshizo Uriu, Shigeyuki Tanaka and Tadashi Wada. *Ibid.*, 1959, **62** (10), 1577–1580.—For the identification of $CH_3SiO_{1\cdot5}$ (**I**), $(CH_3)_2SiO$ (**II**) and $CH_3SiO_{0\cdot5}$ (**III**), characteristic absorptions at 11·7, 12·5 and 13·0 μ, respectively, were used. Since almost all commercial silicones are binary condensation products of **I** and **II**, their quant. analysis can be effected by the following empirical method. When the molar fraction of **I** is <0·6, the ratio of the extinctions measured at 11·7 and 13 μ is expressed by $E_{13}/E_{11\cdot7} = 0\cdot387 + 1\cdot80\ \mathbf{I}/(\mathbf{I} + \mathbf{II})$. When it is >0·6, the relationship is $E_{13}/(E_{13} + E_{12\cdot5}) = 0\cdot485 \times [\mathbf{I}/(\mathbf{I} + \mathbf{II})]^{2\cdot44} + 0\cdot235$. The sample is dissolved in CS_2 to produce a 2% soln. and measured in a 0·1-mm cell. This method was applied to hydrophobic silicones.

III. Determination of the ratio of the methyl to the phenyl radical in silicones. Toshizo Uriu, Shigeyuki Tanaka and Hiroyoshi Yamamoto. *Ibid.*, 1959, **62** (10), 1581–1583.—The following relationship holds between the amounts of methyl and phenyl groups bound to Si—$\log[Me]/[Ph] = 1\cdot507 \log (E_{Me}/E_{Ph}) + 0\cdot202$, where [Me]/[Ph] is the ratio of methyl to phenyl, E_{Me} is the extinction at 3·4 μ, and E_{Ph} is that at 3·3 μ. This method compares favourably with that of Kuratani (J. *Chem. Soc. Japan, Pure Chem. Sect.*, 1952, **73**, 576), which makes use of the C–H stretching at 3·38 μ and the Si–CH_3 vibration at 7·97 μ for the methyl group, and the corresponding vibrations at 3·28 and 6·97 μ for the phenyl group. K. Saito

2055. **Bleached lac.** British Standards Institution (2 Park Street, London, W.1). B.S. 1284:1960. 32 pp.—This standard gives many analytical methods for the determination of the purity of bleached lac.

See also Abstracts—**1819**, Various applications of gas chromatography to org. compounds. **1832**, Water in butanol. **2063**, ^{137}Cs, ^{89}Sr and ^{90}Sr in ion-exchange resins. **2072**, I.r. spectra of carbohydrates in water. **2135**, Determination of isopropamide iodide. **2178**, Nitrocyclohexane in waste water. **2206**, *o*-Hydroxyphenyl in citrus fruits. **2217**, Apparatus for N determination. **2219**, Furnace for org. elementary analysis. **2222**, Detection of amino- and imino-compounds on paper chromatograms. **2223**, Gas chromatography of siloxanes and hydrocarbons. **2232**, Trace impurities (olefins) in hydrocarbons.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE
Biological fluids, animal and vegetable tissues

2056. **Measurement of the specific gravity of urine by the falling-drop method.** K. Davies (Royal Infirmary, Doncaster, England). *J. M. Lab. Technol.*, 1960, **17** (3), 177–178.—A drop urine from a dropping-pipette, constructed so as give a standard-size drop, is allowed to fall through column of immiscible fluid of known specific gravit the rate of fall being timed with a stop-wat Xylene (810 ml) and chloroform (190 ml) is a suitab mixture, with a specific gravity of approx. 0·99 The specific gravity of the urine is obtained fro a graph prepared by timing the rate of fall of dro of standard $CuSO_4$ solutions. R. A. Brenan

2057. **Determination of tritium in urine (liqu scintillation method).** U.K.A.E.A., Producti Group (Windscale, Cumberland, England U.K.A.E.A. Report PG 162 (W), 1960. 6 pp. Full details of the procedure are given. The samp (50 ml) is made slightly alkaline and decolorised boiling with animal charcoal. After re-adjustme of the pH, if necessary, dioxan (9 ml) and a soln. 2,5-diphenyloxazole (4% w/v) and naphthale (10% w/v) in dioxan (9 ml) are added; the mixtu is counted in a scintillation counter, preferably aft passing N through the soln. The content of ^3H derived from this measurement and two others, o on a similar soln. to which a known amount of ^{3} has been added, and another in which the samp is replaced by water. The normal range of conc that can be determined is 0·5 to 100 mμC per m G. J. Hunter

2058. **Rapid assay method for tritium in bacter cells.** E. L. Alpen and H. G. Mandel (Geor Washington Univ. Med. Sch., Washington, D.C U.S.A.). *Biochim. Biophys. Acta*, 1960, **43** (2 317–321.—In the method described, bacteria grow in media containing ^3H compounds are treated wi 10% aq. trichloroacetic acid and then filter through collodion membranes. The membranes a then mounted on aluminium planchettes f counting in a windowless gas-flow counter. I finite-thickness counting values are readily attaine and are convertible to relative total count value The method is considered generally applicable. W. H. C. Shaw

2059. **The preservation of blood for ammon analysis.** A. C. Merchant, R. Goldberger and H. Barker. *J. Lab. Clin. Med.*, 1960, **55** (5), 790–795. Blood obtained by an anaerobic technique in heparinised syringe is introduced under the surfa of mineral oil in a 4-ml Pyrex-glass tube and froze within 10 min. with solid CO_2 and ethanc Subsequent storage in an electric freezer for >24 h causes no change in NH_4^+ concn. W. H. C. Shaw

2060. **Spectrophotometric determination of blo oxygen content.** H. Feinberg and Sister Mary Aln (Michael Reese Hosp., Chicago, Ill., U.S.A.). *Lab. Clin. Med.*, 1960, **55** (5), 784–789.—The bloc (1 ml), sampled and handled anaerobically, is mix with 50% aq. saponin soln. (0·04 ml) and the extin tion is measured at 655 mμ in a 1-mm cell, with spectrophotometer fitted with a photomultiplie and with a narrow slit-width. Total haemoglob is determined separately at 540 mμ (1-cm cell) aft dilution with a cyanmethaemoglobin reagen Blood O is then calculated by the equation give The method is simple, rapid and independent of t haemoglobin concentration, and gives results good agreement with those obtained by the man metric Van Slyke method. W. H. C. Shaw

2061. **Estimation of sulphate in plant materials nd blood.** K. G. Hogan and J. N. Breen (Animal es. Sta., Dept. Agric., Wallaceville, N. Zealand). *.Z. J. Agric. Res.*, 1960, **3** (3), 498–502.—A modified educing mixture for use in the methylene blue olorimetric method of Johnson and Nishita *Anal. Chem.*, 1952, **24**, 736) and a suitable apparatus or use in the routine application of the method are escribed. The proposed reducing mixture is repared by mixing HI (sp. gr. 1·7) (300 ml), 50% $_3PO_2$ (75 ml) and 90% formic acid (150 ml) and oiling in a slow stream of N for 10 min. after the emp. rises to 115°. The method is applicable to a ide range of biological materials and is sufficiently ensitive to determine from 1 to 300 μg of sulphate in 10 to 20 mg of dried pasture or 1 ml of blood. E. C. Apling

2062. **Infra-red analysis of carbon tetrachloride nd ethanol in blood.** R. D. Stewart, T. R. Torkelon, C. L. Hake and D. S. Erley (Dow Chemical Co., fidland, Mich., U.S.A.). *J. Lab. Clin. Med.*, 1960, **6** (1), 148–156.—The sample (5 ml) is extracted by entle shaking with CS_2 (10 ml) for 5 min. and the r. spectrum of the separated CS_2 phase in a 16-mm ell is recorded. Ethanol and CCl_4 may be deterined from the extinctions at 12·8 μ and at 9·55 μ, iving sensitivities of 1 and 10 p.p.m., respectively, n CS_2; CCl_4 is extracted quant., but the results for thanol require correction for only 5% extraction. he method is considered of general application to ne assay of chemicals and metabolites in blood, and ne results obtained on the blood of rabbits exposed o ethanol and CCl_4 are discussed. W. H. C. Shaw

2063. **Determination of caesium-137 and strontium-89 and -90 in milk, vegetation, organic ion-exchange resins, seaweed and fish flesh, and strontium-89 and -90 in bone.** U.K.A.E.A., Production Group (Windscale, Cumberland, England). U.K.A.E.A. Report PG 156 (W), 1960. 1 pp.—Full instructions are given for the procedures. After ignition of the samples the residues are leached with, or dissolved in, HCl; standard radiochemical separations are then used. The assessment of ^{90}Sr is dependent upon the measurement of ^{90}Y. β-Counting is used for all the nuclides. The methods have been applied to samples containing 2 to 1500 $\mu\mu$C of ^{137}Cs, 20 to 5000 $\mu\mu$C of ^{89}Sr, and 1 to 1000 $\mu\mu$C of ^{90}Sr. G. J. Hunter

2064. **Determination of magnesium in body fluids with Titan yellow.** E. Schmid and M. Seibold (Med. Klin., Univ. München, Germany). *Klin. Wochschr.*, 1960, **38** (18), 947–948.—Serum (2 ml) is added to 6 ml of water and the protein is pptd. with 20% (w/v) trichloroacetic acid (2 ml). After centrifugation for 5 min., a 5-ml aliquot of the supernatant liquid is heated at 100° for 2 hr. To the dry residue are added 2 ml of 1% aq. $CaCl_2$ soln., 1 ml of 0·1% aq. poly(vinyl alcohol) and 1 drop of ethanolic methyl red soln., and the mixture is titrated with 0·4N-NaOH to the yellow end-point. Titan yellow soln. (0·04%) (2 ml) is then rapidly added, followed by 5 ml of 2N-NaOH, and the volume is made up to 25 ml with water. The extinction is measured, in 5-cm cells, with a S55E filter, and is compared with that of standard soln. containing 0 to 50 μg of Mg per ml. For whole blood, or red blood cells, a 1-ml aliquot is haemolysed with 7 ml of water, the proteins are pptd. and removed, and the Mg is determined as for serum. For urine, a 10-ml aliquot is shaken with 0·6 g of powdered charcoal, and the soln. is filtered, a suitable aliquot being used for the determination as described above. There is no interference from Na, K, Ca, Zn, Fe or Cu. Values reported are in good agreement with those obtained by other workers. D. B. Palmer

2065. **Micro-determination of phosphorus in biological substances. Modification of the method of Zinzadse.** G. J. M. Hooghwinkel and H. P. G. A. van Niekerk (Dept. of Med. Chem., Univ., Leyden, Netherlands). *Proc. Kon. Ned. Akad. Wet., B*, 1960, **63** (4), 475–483 (in English).—*Procedure for inorganic P*—To the nearly neutral soln. (2 to 20 or 5 to 50 μg of P) in a constricted test-tube calibrated at 10 (or 25) ml add 2 drops of 0·1% 2,4-dinitrophenol indicator soln. in ethanol and 0·2 (or 0·5) ml of Zinzadse reagent (*Z. PflErnähr.*, 1930, **15**, 129). Dilute to the mark with water and immerse the tube in a boiling-water bath for 30 min. Cool, re-adjust the vol. to the mark and measure the extinction at 825 mμ in a 10-mm cell. Carry out the procedure on a blank and standard soln. in the same way. *For organic P*—Heat the sample (2 to 20 μg of P) with $HClO_4$ (0·5 ml) and a few drops of conc. H_2SO_4 in a Kjeldahl flask for 20 min. Cool, neutralise with 5N-NaOH to 2,4-dinitrophenol indicator and proceed as for inorganic P. Beer's law is obeyed for concn. up to 50 μg of P; the colour is stable for several days and the reagent for more than 4 yr. The sensitivity is high, 0·34 p.p.m. of P giving 50% transmission. The Zinzadse reagent is a soln. of MoO_3 in H_2SO_4 reduced with metallic molybdenum, and, by its use, the need for reduction after reaction with the phosphate is avoided. J. I. M. Jones

2066. **Simple micro-determination of phosphoric acid in biological material after Kjeldahl decomposition by the molybdate - vanadate method.** G. Pulss (Inst. f. Tierphysiol. u. Tierernährung. Univ., Hospitalstrasse **34**, Kiel). *Z. anal. Chem.*, 1960, **176** (6), 412–421 (in German).—The method described is simple and insensitive to interference. Nitrogen and phosphoric acid may be determined in a Kjeldahl digest. Inorganic phosphate can be determined in citrated and oxalated plasma and in enzyme preparations. *Construction of calibration curve*—Known amounts of KH_2PO_4 soln. (containing 0·2 to 9 μg of P per ml) are transferred to 50-ml flasks and 10 ml of H_2SO_4 (1:10) and 10 ml of reagent [0·25% ammonium vanadate soln. - 10% ammonium molybdate soln. (1:1)] are added. The soln. are mixed and made up to vol. After 5 min. the extinctions are measured in a 2-cm cell against a blank in an Eppendorf photometer with a Hg 405 mμ filter, and the results are plotted. Procedures are described for the determination of inorganic phosphate in plasma or serum, of pyrophosphate, of total phosphate in whole blood and plasma, and of nitrogen and total phosphate in a Kjeldahl digest. A. C. R. Hartley

2067. **Micro-determination of gold in biological fluids employing chloric acid and o-tolidine.** J. F. Goodwin and A. J. Bollet (Wayne State Univ. Coll. Med., Detroit, Mich., U.S.A.). *J. Lab. Clin. Med.*, 1960, **55** (6), 965–972.—Samples of serum or plasma (1 to 3 ml), urine (10 to 50 ml), tissue or faeces (0·3 to 0·7 g dry wt.) are digested with a chloric acid reagent, and the residue is neutralised and treated with aqua regia. After the addition of aq. KHF, the soln. is treated with an *o*-tolidine reagent, and the stable colour is measured at 427 mμ after 20 min. Standards containing 1 to 8 μg of Au and blanks of

the corresponding Au-free samples are treated similarly. Calibration is rectilinear.

W. H. C. Shaw

2068. **Fluorimetric determination of uranium in biological material.** U.K.A.E.A., Production Group (Springfields, Lancs.). U.K.A.E.A. Report PG 143 (S), 1960. 12 pp.—Full details are given of a procedure that has been applied to lung, kidney, thyroid, liver, spleen, lymph nodes and bone. Soft tissue is treated with HNO_3 and after evaporation of the soln. the residue is ignited and leached with HNO_3; bone samples are ashed and dissolved in HNO_3. After the addition of soln. of $Ca(NO_3)_2$ and $Al(NO_3)_3$ the U is extracted into ether, which is then distilled off. The residue is fused with NaF and the fluorescence of the cooled melt is measured; a known amount of U is then added and, after re-melting the NaF, the fluorescence is measured again; the content of U is derived from the two readings.

G. J. Hunter

2069. **The analysis of plutonium-241 in urine.** J. J. D. Ludwick (Gen. Electric Co., Hanford Atomic Products Operation, Richland, Washington). *U.S. Atomic Energy Comm.*, Report HW-64170, 1960. 9 pp.—A method was developed for the quantitative determination of ^{241}Pu in exposed personnel through urine analysis. The procedure was designed for use, when necessary, in conjunction with the Hanford plutonium bio-assay technique of alpha analysis. The method is based on the recovery of Pu from electro-deposited discs and subsequent extraction of the Pu from N-HCl into a liquid scintillator containing dibutyl phosphate. The ^{241}Pu β-emission is detected in a standard liquid scintillation spectrometer. The yield for Pu recovery by using spiked samples of ^{241}Pu was $85 \pm 9\%$. Samples containing as little as $2{\cdot}2 \times 10^{-6}$ μC of ^{241}Pu may be reliably detected. This represents 3·5% of the max. permissible dose 90 days after personnel exposure.

Nucl. Sci. Abstr.

2070. **Colorimetric method for the determination of Resochin (chloroquine phosphate) [in urine].** R. Strufe (Chemotherap.-Inst. d. Farbenfabriken Bayer A.-G., Werk Wuppertal-Elberfeld, Germany). *Clin. Chim. Acta*, 1960, **5** (5), 753–756.—A red colour is produced by chloroquine (**I**) in 1,2-dichloroethane with a reagent consisting of a 0·4% soln. of *trans*-aconitic acid in acetic anhydride; 5 μg of **I** can be detected. To determine **I** in urine, add 0·1N-NaOH (0·5 ml) to an aliquot (5 ml) of a 24-hr. sample and shake the mixture with 1,2-dichloroethane (10 ml) for 30 sec. Centrifuge, separate the organic solvent and wash it by shaking with 0·1% $NaHCO_3$ soln. (10 ml). Filter the solvent through a small filter-paper and to 5 ml add the reagent soln. (0·1 ml). After 20 min. at room temp. measure the colour at 570 mμ. Prepare a standard curve from normal urine samples containing added **I** (0 to 100 μg per 5 ml of urine).

H. F. W. Kirkpatrick

2071. **Determination of "carboxytolbutamide" in urine.** E. Nelson, I. O'Reilly and T. Chulski (Sch. of Pharm., Univ. of Calif. Med. Centre, San Francisco, U.S.A.). *Clin. Chim. Acta*, 1960, **5** (5), 774–776.—To suitably diluted urine (3 ml) containing 2 to 10 mg of "carboxytolbutamide" (*N*-butyl-*N'*-*p*-carboxybenzenesulphonylurea) (**I**) per 100 ml add 5N-HCl (1 ml) and amyl acetate (6 ml), stopper the tube and gently turn it through 180 50 to 70 times. Centrifuge, withdraw 4 ml of th acetate layer and add 0·1% fluorodinitrobenzen soln. in amyl acetate (1 ml). Place the tube in tube heater at 150° ± 1° for exactly 5 min., coo and read the absorption at 380 mμ. Construct standard curve by substituting 3 ml of soln. con taining 2 to 12 mg of **I** per 100 ml for the dilute urine. Tolbutamide may be removed before thi procedure, if its presence is suspected, by tw extractions with $CHCl_3$ at pH 5·5. Normal tes subjects excreted little or no intact tolbutamid after ingestion of **I**.

H. F. W. Kirkpatrick

2072. **Infra-red spectra of carbohydrates in wate and a new measure of mutarotation.** F. S. Parke (New York State Univ., Brooklyn, U.S.A.). *Bio chim. Biophys. Acta*, 1960, **42** (3), 513–519.—Th infra-red absorption curves of aq. solutions (10 t 20% w/v) of nine carbohydrates were studied an shown to be characteristic for each compound. Th extinctions at suitable wavelengths for α-D-glucose β-D-glucose and for β-D-mannose are shown t change with time because of mutarotation, and th mutarotation constants derived by this metho agree well with those determined polarimetrically

W. H. C. Shaw

2073. **Detection of reducing sugars in urin specimens by paper chromatography.** S. N. Tewar (18/3A, The Mall, Kanpur, India). *Z. anal. Chem.* 1960, **176** (6), 406–408 (in English).—The urin (0·005 to 0·02 ml, according to the sugar concn.) i spotted on Whatman No. 1 paper; standard suga soln. are also applied, 2 to 3 cm apart. Chromato grams are developed by the ascending techniqu with suitable solvents, and the spots are revealed with 1,3-dihydroxynaphthalene, benzidine or aniline diphenylamine reagents, by the dipping technique R_F values are quoted, and colours given by the thre reagents, for nine sugars with four solvents. Gener ally, not more than four sugars are present in an sample.

A. C. R. Hartley

2074. **Glucose detection in urine.** Miles Labora tories, Inc. [Inventor: H. M. Free]. Brit Pat 855,013, date appl. 31.12.56.—The composition fo detecting glucose in urine (*cf. Anal. Abstr.*, 1959 **6**, 3099), which comprises a mixture of glucos oxidase, peroxidase and a compound which under goes a colour change in the presence of hydroge peroxide, *e.g.*, *o*-tolidine dihydrochloride, is stabilise by addition of a sol. protein material, *e.g.*, plasma serum, albumin or gelatin.

J. M. Jacobs

2075. **Enzymatic estimation of erythrocytic galac tose 1-phosphate.** H. N. Kirkman and E. S. Maxwel (Nat. Inst. Health, Bethesda, Md., U.S.A.). *J Lab. Clin. Med.*, 1960, **56** (1), 161–166.—The metho is based on the catalysis by galactose 1-phosphat uridyl transferase from haemolysed normal huma erythrocytes of the reaction between galactose 1 phosphate (**I**) and uridine diphosphoglucose (**II**) t give uridine diphosphogalactose and glucose 1 phosphate. Unchanged **II** is determined from th change in extinction at 340 mμ resulting from th oxidation of **II** to uridine diphosphogluconic acid i the presence of diphosphopyridine nucleotide. Con centrations of **I** up to 35 mg per 100 ml can b determined with an error for the mean of duplicate of ± 1·7 mg per 100 ml. The uses of the method fo the detection of galactoseaemia and for dietar control are discussed.

W. H. C. Shaw

2076. **Estimation of fucose in saliva.** D. A. P. ans (Liverpool Univ., England). *J. Lab. Clin. d.*, 1960, **55** (3), 381–385.—An adaption of the oglycollic acid - H_2SO_4 method of Gibbons *nal. Abstr.*, 1955, **2**, 2465) for methylpentoses is scribed. A spectrophotometric procedure for the tection of interfering substances is given and a ndard error of $\simeq 1\%$ is attained. The fucose els obtained, and their significance in relation to od-group substances, are discussed.
W. H. C. Shaw

2077. **Identification of acid mucopolysaccharides m granulation tissue in rats.** G. S. Berenson and R. Dalferes (State Univ. School of Med., New leans, La., U.S.A.). *Brit. J. Exp. Path.*, 1960, (4), 422–429.—Defatted tissue from artificially-luced granulation tissue was extracted with kali, hydrolysed with trypsin and deproteinised th trichloroacetic acid. The mucopolysaccharides re pptd. with ethanol and characterised by zone ctrophoresis and column chromatography. On e basis of the behaviour of the extracts on hydro-sis with hyaluronidase, and on infra-red spectral alysis, and of the following characteristics—tical rotation and nitrogen, uronic acid, hexos-ine and sulphate contents—four fractions were entified as hyaluronic acid, heparitin sulphate, ondroitin sulphate A and chondroitin sulphate B.
D. E. Evans

2078. **Paper-chromatographic determination of keto-acids in blood and urine.** E. Zelníček nst. Med. Chem., Med. Fac. Univ., Brno, Czecho-ovakia). *Čas. Lék. Čes.*, 1960, **99** (29), 927–928.—he keto-acids are converted into the corresponding nitrophenylhydrazones and separated chromato-aphically. *Procedure*—Deproteinise the blood mple with tungstate and H_2SO_4 and proceed cording to Neish (*Rec. Trav. Chim. Pays-Bas*, 953, **72**, 105). Dissolve the ethereal residue in hyl acetate and place on Whatman No. 1 paper aliquot of the dinitrophenylhydrazones equiva-nt to 0·5 ml of blood or urine. Chromatograph the descending technique with n-butanol - hanol (96%) - aq. NH_3 (1%) (6:1:3). After 12 14 hr. elute the dinitrophenylhydrazones with N-a_2CO_3 and determine spectrophotometrically at 80 mμ. The R_F values of the separated compounds ere determined.
J. Žýka

2079. **A method for the determination of 4-ydroxy-3-methoxymandelic acid ("vanilmandelic cid") for the diagnosis of phaeochromocytoma.** . W. Sunderman, jun., P. D. Cleveland, N. C. Law nd F. W. Sunderman (U.S. Naval Med. Sch., ethesda, Md., U.S.A.). *Amer. J. Clin. Path.*, 1960, **4** (4), 293–312.—4-Hydroxy-3-methoxymandelic cid (**I**) is extracted from urine with ethyl acetate, fter adsorption of impurities with magnesium licate. The extract is oxidised to vanillin with $K_3Fe(CN)_6$ in the presence of Zn at an acid pH. he vanillin is determined photometrically after ddition of an indole - phosphoric acid colour eagent. Recovery of **I** averages 94% ± 7·6. In ormal and hypertensive subjects, excluding atients with phaeochromocytoma, the excretion f **I** ranged from 0·7 to 6·8 mg per 24 hr. In 3 ases of benign phaeochromocytoma, the values ere 15, 38 and 41 mg per 24 hr. In a case of alignant phaeochromocytoma, the value was 50 mg per 24 hr. It is suggested that this deter-ination has some advantages over the determina-on of catecholamines in the diagnosis of phaeo-romocytoma.
D. E. Evans

2080. **Effect of borate on the carbazole reaction [for uronic acids].** J. D. Gregory (Rockefeller Inst., New York, U.S.A.). *Arch. Biochem. Biophys.*, 1960, **89** (2), 157–159.—The colour developed in the determination of certain uronic acids by Dische's carbazole method (*cf. J. Biol. Chem.*, 1947, **167**, 189) is increased by the addition of borate, which modifies the formation of intermediates during heating with H_2SO_4. The mechanism of the reaction is discussed.
W. H. C. Shaw

2081. **Determination of flavonoids by chromatography on polyamide powder.** J. Davídek (Zentralforschungsinst. f. Lebensmittelindustrie, Prague, Czechoslovakia). *Nahrung*, 1960, **4** (7), 661–666.—After establishing that the adsorption on and elution of flavonoids from a polyamide-powder column are quantitative, a method has been developed for the determination of flavonoids in natural substances (elder flowers and buckwheat). *Procedure*—The dried sample (2 to 15 g) is extracted in a Soxhlet apparatus for 8 hr. with methanol. The extract is evaporated to one-third of its volume and diluted with an equal volume of water. The ppt. of chlorophyll is filtered off and the filtrate is made up to 100 ml with water. This soln. is used for chromatography. The column is washed and eluted with methanol (30%), the eluate is received in a 25-ml flask, and made up to vol., and 1 ml is used for the colorimetric determination. The accuracy is ±10%.
I. Dickinson

2082. **Determination of long-chain fatty acid esters in serum.** Seiji Kasuga (Kanto-Teishin Hosp., Gotanda, Shinagawa, Tokyo, Japan). *Clin. Chim. Acta*, 1960, **5** (5), 772–773.—A Bloor extract of serum is treated after evaporation of the ether with alkaline hydroxylamine soln. After acidification the hydroxamic acids are extracted with xylene and treated with alkaline ferric reagent to give ferric hydroxamate, the colour of which is read at 425 mμ. Methyl palmitate is used as a standard. By this method increased stability of colour is obtained.
H. F. W. Kirkpatrick

2083. **Silicon tetrachloride-treated paper for the chromatography of phosphatides. I. Preparation, properties and application.** H. G. Bungenberg de Jong and J. T. Hoogeveen (Univ. Leyden, Netherlands). *Proc. Kon. Ned. Akad. Wet., B*, 1960, **63** (2), 190–205 (in English).—Two varieties of $SiCl_4$-treated paper are described (*i*) acid paper, and (*ii*) neutralised paper treated with NH_3 vapour. Their use for the separation of phosphatides is compared with silicic acid-treated paper.

II. Mechanism of the chromatography of egg phosphatides. H. G. Bungenberg de Jong and J. T. Hoogeveen. *Ibid.*, 1960, **63** (3), 228–257.—Further studies in the use of this paper have been made. Egg phosphatides give four spots: cephalin, lecithin, lysocephalin plus sphingomyelin (not separated), and lysolecithin.

III. Comparison between chromatography of egg phosphatides on silicon tetrachloride-treated paper and on non-impregnated paper. Role of the gradient in the mobile phase and influence of adsorbent. H. G. Bungenberg de Jong and J. T. Hoogeveen. *Ibid.*, 1960, **63** (4), 383–409.—The mechanism of the chromatography of egg phosphatides is studied with pure lecithin and mixtures of lecithin, cephalin, lysolecithin, lysocephalin and sphingomyelin. Studies were made (*a*) by placing 9 spots of the same concn. along a line running diagonally across the chromatographic paper, and (*b*) by placing spots of

different concn. along a horizontal starting line, running ascending chromatograms and staining with acid fuchsine - uranyl nitrate - 0·01N-HCl (Jong and Someren, *Anal. Abstr.*, 1960, **7**, 217; Hooghwinkel *et al.*, *Anal. Abstr.*, 1960, **7**, 1498). In (*a*) the developed spots lay along an inverted logarithmic curve on both untreated and $SiCl_4$-treated paper, suggesting a concn. gradient in the mobile phase, confirmed experimentally. In (*b*) the size and shape of the spots varied with the concn., those of low concn. being small, circular and uniformly stained, and those of high concn. being triangular, large and with increasing colour intensity in the upward direction. It is concluded that the mechanism is one of adsorption chromatography by gradient elution. Separation of mixtures was incomplete, but differed according to whether the paper used was treated or not. Microscopical and chemical studies point to the adsorbent in the treated paper being SiO_2 deposited as a sheath around the cellulose fibres, the concn. of $SiCl_4$ used having no substantial effect on the separation on neutralised paper. A slightly increased separation on non-neutralised paper with increasing $SiCl_4$ concn. is attributed to HCl produced by the reaction of the $SiCl_4$ with moisture in the paper.

J. I. M. Jones

2084. **Quantitative paper chromatography of phosphatides. The acid - base type phosphatides of hen's egg-yolk and human aorta.** G. J. M. Hooghwinkel and H. P. G. A. van Niekerk (Dept. of Med. Chem., Univ., Leyden, Netherlands). *Proc. Kon. Ned. Akad. Wet., B*, 1960, **63** (4), 469–474 (in English).—Extract the yolk of boiled eggs or the aorta with methanol - $CHCl_3$ (2:3). Evaporate to dryness, re-dissolve the residue in methanol - $CHCl_3$ (1:4) to 4% concn. and carry out ascending chromatography on S. & S. paper 2043b impregnated with $SiCl_4$ (Jong and Someren, *Anal. Abstr.*, 1960, **7**, 217; Hooghwinkel *et al.*, *Anal. Abstr.*, 1960, **7**, 1498). Stain the chromatogram with 0·005% Edicol Supra Ponceau 4RS (C.I. Food Red 7), and 0·2% uranyl nitrate soln. in 0·01N-HCl. Cut out the stained spots and elute separately with 50% t-butyl alcohol in 0·6N-HCl and measure the extinction at 510 mμ. The spots correspond to the following phosphatides, the molecular percentages in egg yolk and human aorta, respectively, being given in parentheses—phosphatidylethanolamine (14·9; 13·8), phosphatidylcholine (72·5; 29·5), sphingomyelin (6·4; 52·5) and lysophosphatidylcholine (6·2; 4·4). The reproducibility is high and the accuracy is within 1%.

J. I. M. Jones

2085. **Determination of amino-sugars in the presence of amino-acids and glucose.** C. Cessi and F. Piliego (Ist. di Patolog. Gen., Univ. Bologna, Italy). *Biochem. J.*, 1960, **77** (3), 508–510.—The method is based on the observation of Schloss (*Anal. Chem.*, 1951, **23**, 1321) that amino-sugars yield volatile chromogens when heated with acetylacetone in alkaline soln. The volatile fraction is separated by distillation, and a red colour is formed by reaction with *p*-dimethylaminobenzaldehyde. The extinction is determined at 545 mμ. The coeff. of variation is 2·4%. Only methylamine interferes, and amino-acids in the presence of glucose give only non-volatile chromogens.

J. N. Ashley

2086. **Catecholamines and the diagnosis of phaeochromocytoma.** R. Straus and M. Wurm (Dept. of Path., St. Joseph Hosp., Burbank, Calif., U.S.A.). *Amer. J. Clin. Path.*, 1960, **34** (5), 40 425.—A review, with 184 references, is present which includes a discussion of chemical and biologi methods of determination of catecholamines body fluids. D. E. Evans

2087. **Mass-spectrometric identification of amir acids.** K. Biemann, J. Seibl and F. Gapp. *Bioche Biophys. Res. Commun.*, 1960, **1**, 307–311.—T application of mass spectrometry to the identifi tion, separation and structure determination amino-acids is described. The spectra of the et esters of several amino-acids were obtained a interpreted. By this method it is also possible identify the components of a complex amino-a mixture without prior separation.

Brit. Gel. Glue Res. Ass. Abstr.

2088. **Analysis of mixtures of amino-acids by g chromatography.** N. Melamed and M. Rena (Lab. de Chim. Gén. et Org., Inst. Agron. de l'Ét Gembloux, Belgium). *J. Chromatography*, 19 **4** (4), 339–346 (in French).—Treatment of α-amir acids with conc. HCl and HNO_3 yields the corr ponding α-chloro-acids, the methyl esters of whi can be separated by gas chromatography at 13 on a composite column consisting of 2 metres polyoxyethylene glycol and 2 metres of silico oil - stearic acid. Good separations of the est prepared from mixtures of glycine, alanine, aminobutyric acid, valine, norvaline, leucir isoleucine and norleucine have been obtained.

R. M. Rowley

2089. **Colorimetric ninhydrin method for to alpha-amino-acids in urine.** A. Khachaduria W. E. Knox and A. M. Cullen (Harvard Med. Sc Boston, Mass., U.S.A.). *J. Lab. Clin. Med.*, 19 **56** (2), 321–332.—In a rapid simple adaption of t colorimetric ninhydrin method, no interference encountered from NH_3, urea, bound amino-aci or traces of protein. Glycine is used as standa and values equivalent to 80% of the total fr α-amino-acid nitrogen are obtained with an error ±5%. Little error is introduced by the differe amino-acid composition of different samples, a the method is suitable for the detection of modera and gross amino-aciduria. W. H. C. Shaw

2090. **Application of Edman's phenyl isothi cyanate method in the determination of C-termin amino-acids and peptides after hydrazinolys** P. Wallén and I. Sjöholm (Chem. Dept. II, Karoli ska Inst., Stockholm, Sweden). *Acta Che Scand.*, 1960, **14** (8), 1749–1762 (in English). The hydrazinolysis procedure of Akabori *et a* (*Bull. Chem. Soc. Japan*, 1952, **25**, 214) has be combined with the phenyl isothiocyanate meth for the identification and quant. determination C-terminal amino-acids and peptides. A new solve system is described for the separation by pap chromatography of the phenylthiohydantoin derivatives of lysine and ornithine (the latter formed from arginine on hydrazinolysis). Th method is applied to fibrinopeptides A and B a to insulin. Some peptide bonds (valine - arginin alanine - arginine, proline - lysine) are found to resistant to hydrazinolysis. *Procedure*—Weig the dried material (3 to 55 mg) into 4-ml test-tub fitted with ground glass stoppers. Add anhy hydrazine (prep. described) (0·1 to 0·5 ml) a heat the stoppered tubes for 10 hr. at 100°. Remov the excess of hydrazine *in vacuo* over H_2SO Dissolve the residue in 0·5 ml of H_2O, shake 3 tim

ı 0·75 ml of n-heptane - n-heptaldehyde (1:1) 5, 30 and 30 min., then twice with n-heptane ml) and twice with n-pentane (0·5 ml). Dry aq. phase *in vacuo* over P_2O_5 and store at −15°. pare the **I** derivatives by the procedure of nan (*Acta Chem. Scand.*, 1950, **4**, 283) as modified Sjöquist (*Biochim. Biophys. Acta*, 1960, **41**, 20). the separation of the ornithine and lysine ivatives, use the organic phase of n-heptane - ıloroethane - 75% formic acid (1:1:2). The ine and alanine derivatives are also well ırated in this system. When a preliminary ıration of peptides is required, electrophoresis arried out on Whatman 3MM paper in a buffer ı. (pH 4·9), containing 6 ml of glacial acetic acid 8 ml of pyridine per litre of water, for 1·5 hr. 25 V per cm. The fractions are located on a rker strip with ninhydrin or by u.v. fluorescence, the main fractions are eluted with water and porated to dryness *in vacuo* over P_2O_5. The tides are then hydrolysed with constant-boiling l at 105° for 23 hr. in evacuated sealed tubes, the amino-acids are separated by two-dimen- ıal paper chromatography with the upper phase n-butanol - acetic acid - water (4:1:5) for the t direction (descending) and pyridine - water l) for the second direction (ascending). The ınt. analysis is then carried out by the method of quist (*loc. cit.*). G. Russell

2091. **Colorimetric determination of arginine ester drolysis by human sera.** M. E. Brown (Faulkner sp., Boston, Mass., U.S.A.). *J. Lab. Clin. Med.*, 60, **55** (4), 616–624.—The sample (0·5 ml) is ubated for 1 hr. with barbiturate buffer (1 ml) l substrate (1 ml) containing 20 μmoles of enzoyl-L-arginine ethyl ester. Proteins are then d. with trichloroacetic acid and removed by trifugation. The supernatant liquid is treated h alkaline hydroxylamine and the hydroxamic ds resulting from the ester remaining after ubation are determined colorimetrically with aq. $FeCl_3$. The method is applied to the study of serum zymes, and kinetic aspects are discussed.
W. H. C. Shaw

2092. **Application of the Sakaguchi reaction to determination of dinitrophenylarginine.** W. nson (Hannah Dairy Res. Inst., Ayr, Scotland). *chim. Biophys. Acta*, 1960, **44** (1), 182–184.— e dinitrophenylarginine obtained in the study of terminal amino-acids of proteins by the dinitro- enylation procedure may be determined satis- torily by the 1-naphthol - hypobromite reaction ovided that the standardised conditions of the dified method described are adopted and that standard employed is of the highest purity. e working range is from 50 to 500 μg and cali- ation is rectilinear. W. H. C. Shaw

2093. **Enzymatic spectrophotometric method for determination of phenylalanine in blood.** B. N. Du and P. J. Michael (Nat. Inst. Health, thesda, Md., U.S.A.). *J. Lab. Clin. Med.*, 1960, (3), 491–496.—The method described is based on measurement of the extinction of the enol - rate complex of phenylpyruvic acid generated m phenylalanine by snake venom L-amino-acid idase. Tyrosine and tryptophan interfere but y be corrected for and determined by a three- nt spectrophotometric procedure. The method apid, precise and accurate for the determination elevated phenylalanine levels in sera, and for the tection of phenylketonuria, and gives results in good agreement with those obtained by other methods. W. H. C. Shaw

2094. **Identification of 5-aminovaleric acid in the urine of rats injected with cadaverine.** C. De Marco and B. Mondovi (Ist. Chim. Biol., Univ., Roma). *Ital. J. Biochem.* (English Edition), 1960, **9** (4), 226–232.—5-Aminovaleric acid (**I**) and cadaverine (pentamethylenediamine) (**II**) were identified in the urine of adult male rats injected subcutaneously with 60 mg of **II** dissolved in 2 ml of H_2O and neutralised. The urine (24-hr. sample) was diluted to 25 ml with H_2O, decolorised with charcoal and de-salted; 0·2 ml was subjected to two-dimensional chromatography with (*i*) phenol - H_2O in an NH_3 atmosphere, and (*ii*) collidine - lutidine - H_2O. Subsequent re-chromatography of the spots of **I** with butanol - acetic acid - H_2O confirmed that no 4-aminobutyric acid was present. Spots were revealed with ninhydrin. J. I. M. Jones

2095. **Colorimetric method for determination of erythrocyte glutathione.** T. D. Stevenson, B. L. McDonald and S. Roston (Louisville Univ. Med. Sch., Ky., U.S.A.). *J. Lab. Clin. Med.*, 1960, **56** (1), 157–160.—The method described is based on the reaction of di-(*p*-nitrophenyl) disulphide with thiols to yield the stable yellow nitrobenzene-thiol anion. A description is given of the clinical application of the method to the detection of erythrocyte sensitivity to haemolysis by certain drugs, which is related to the diminished content of reduced glutathione after incubation of the erythrocytes with acetylphenylhydrazine. Good correlation of results is attained with those given by the nitroprusside method. W. H. C. Shaw

2096. **Quantitative staining of isolated serum-protein fractions of normal and pathological sera.** E. Roller, G. Berg and F. Scheiffarth (Med. Univ.-klinik, Erlangen, Germany). *Clin. Chim. Acta*, 1960, **5** (5), 695–701.—Staining with Amido black 10B is investigated. Identical fractions of different sera can show differences in staining, and different fractions of one serum are also stained differently. The causes of these differences are discussed.
H. F. W. Kirkpatrick

2097. **Determination of total protein in spinal fluid with sulphosalicylic acid and trichloroacetic acid.** O. Meulemans (Dept. of Pediat., State Univ., Utrecht, The Netherlands). *Clin. Chim. Acta*, 1960, **5** (5), 757–761 (in English).—Two methods are recommended. (*i*) To 1 ml of fluid add 4 ml of a mixture of equal vol. of 6% aq. sulphosalicylic acid soln. and 14% aq. Na_2SO_4 soln. After 10 min. read the turbidity at 450 mμ. (*ii*) To 1 ml of fluid add 4 ml of a 3% aq. soln. of trichloroacetic acid. Measure the turbidity at 450 mμ after 10 min., re-mixing before reading. In both methods the factor for converting the reading to protein (mg per 100 ml) is obtained by treating diluted serum of known protein content similarly. These methods give results comparable with the biuret method. The results with 3% sulphosalicylic acid soln. are unsatisfactory. H. F. W. Kirkpatrick

2098. **Chromatography of polyelectrolytes. VI. Protein chromatography on ion-exchange cellulose derivatives.** G. Semenza (Biochem. Inst., Univ., Zurich). *Chimia*, 1960, **14** (10), 325–343 (in German). —A review, with 59 references and an extensive table of published applications. R. E. E.

2099. **Quantitation of gamma-globulins in human sera by immuno-precipitation.** B. Barrett, P. A. Wood and W. Volwiler (Washington Univ. Med. Sch., Seattle, U.S.A.). *J. Lab. Clin. Med.*, 1960, **55** (4), 605–615.—The method described is based on the use of human-γ-globulin anti-serum obtained from rabbits. The serum sample and anti-serum are allowed to react under standard conditions and the ppt. formed is separated by centrifugation, washed twice by centrifuging and then determined by a modified Folin method. The amount of γ-globulin in the sample is obtained by reference to a standard calibration curve prepared for each batch of anti-serum. Cross-agglutinating antibodies in the anti-serum are removed by the stepwise addition of hypo-γ-globulinaemic serum. Levels as low as 1 to 5 mg of γ-globulins per 100 ml may be determined satisfactorily. Results are compared with those obtained by electrophoresis, and the differences are discussed.

W. H. C. Shaw

2100. **Use of fractionated deproteinisation of serum for the determination of the soluble protein fractions.** S. Turek (Inst. of Hygiene, Charles' Univ., Prague, Czechoslovakia). *Clin. Chim. Acta*, 1960, **5** (5), 689–694 (in German).—In the method described, flocculation by heat of heat-denaturable serum proteins and final deproteinisation with $HClO_4$ are used. The results are compared with those by direct deproteinisation with $HClO_4$, and differences observed between normal and pathological sera are discussed.

H. F. W. Kirkpatrick

2101. **Separation of plasma glucoproteins by paper electrophoresis. I. Technique and results in normals.** F. Infante, S. Juillard, D. Schwartz and M. Sanz (Hôp. Cantonal, Genève, Switzerland). *Clin. Chim. Acta*, 1960, **5** (5), 672–679 (in French).—A modified staining method is proposed in which the dimethyl derivative of fuchsine (Neufuchsine) is used for the HIO_4 - Schiff reaction, and potassium metabisulphite as reducing agent. Results are obtained by modified reading methods.

H. F. W. Kirkpatrick

2102. **Laboratory and clinical evidence of the reliability of the alkaline incinerator method of serum-protein-bound iodine measurement.** B. A. Walter, R. J. Henry, A. G. Ware and P. Starr (Los Angeles County Hosp., Calif., U.S.A.). *J. Lab. Clin. Med.*, 1960, **55** (4), 643–649.—A statistical and clinical study is presented of results obtained for protein-bound iodine by the colorimetric determination of iodine with Ce^{4+} after alkaline incineration. The method is considered reliable for routine clinical use in the diagnosis and treatment of thyroid disease.

W. H. C. Shaw

2103. **Further modification of the benzidine method for measurement of haemoglobin in plasma.** G. E. Hanks, M. Cassell and H. Chaplin (Washington Univ. Med. Sch., St. Louis, Mo., U.S.A.). *J. Lab. Clin. Med.*, 1960, **56** (3), 486–498.—A tenfold increase in sensitivity of the benzidine - H_2O_2 method for haemoglobin in plasma is attained in the method described. Details are given for the purification of the benzidine and for the preparation of a stable reagent. Haemolysis of the sample during manipulation is minimised by the technique adopted, and the precautions necessary for accurate results are studied. The mean normal value is 0·31 mg of haemoglobin (range 0·16 to 0·58) per 100 ml of plasma.

W. H. C. Shaw

2104. **Standardisation of haemoglobinometry. The extinction coefficient of haemoglobin cya (cyanomethaemoglobin).** W. G. Zijlstra and E van Kampen (Inst. of Physiol., Univ. of Grönig The Netherlands). *Clin. Chim. Acta*, 1960, **5** 719–726 (in English).—The method introduced Stadie (*J. Biol. Chem.*, 1920, **41**, 237) is mad basis for the standardisation. The extinction co at 540 mμ of the cyanomethaemoglobin is establis as 11·0.

H. F. W. Kirkpatric

2105. **Ultra-violet absorption of palladium determination of carbon monoxide haemoglo** L. A. Williams, R. A. Linn and B. Zak (Wa State Univ. Coll. of Med., Detroit, Mich., U.S. *Amer. J. Clin. Path.*, 1960, **34** (4), 334–337.— this method, the CO is passed into a soln. contain excess of Pd^{2+}; the soln. is diluted to a fixed with HCl and the extinction of the soln. is measu at 278 mμ. No colour reagent is therefore requi

D. E. Evan

2106. **Substances interfering with spectroph metric estimation of nucleic acids and their elimi tion by the two-wavelength method.** R. Tsa and G. G. Markov (Method. Inst. Biol., Acad. Sci., Sofia, Bulgaria). *Biochim, Biophys. A* 1960, **42** (3), 442–452.—From a study of the opt properties of substances interfering with the determination of ribo- and deoxyribo-nucleic ac in tissue extracts, it is concluded that satisfact correction can be achieved by measuring extinctions at 260 and 286 mμ and at 268 284 mμ for the respective nucleic acids. Res are then calculated by the procedures describ A comparison is made with results obtained by ot methods.

W. H. C. Shaw

2107. **Separation of inosine and adenosine p phosphate nucleotides by paper chromatogra** P. Cerletti (Ist. Chim. Biol., Univ., Roma). *Ital Biochem.* (English Edition), 1960, **9** (4), 237–24 Good separation of inosine and adenosine nuc tides is obtained by descending chromatography Munktell CHR 100 paper in 6 hr. with 16% amm ium bicarbonate soln. The spots are located photographed in u.v. light by the procedure Markham and Smith (*Biochem. J.*, 1951, **49**, 4 No hydrolysis takes place during separation, elution of the spots with H_2O at 0·5 ml per (Brimley and Barrett, "Practical Chromatograph Chapman and Hall, London, 1953, p. 37) perm the quant. estimation by the determination ribose (Albaum and Umbreit, *J. Biol. Chem.*, 19 **167**, 369); 5 ml of eluate suffices for comp recovery. The R_F values are—inosine triphosph 0·94; inosine diphosphate, 0·88; inosine mo phosphate, 0·84; adenosine triphosphate, 0· adenosine diphosphate, 0·80; adenosine mo phosphate, 0·75; inosine, 0·71; and adenosine, 0 Further separation is obtainable by two-dimensio chromatography with n-propanol - trichloroac acid - H_2O - aq. NH_3 (22° Bé) (75:5:20:1) in direction and ammonium bicarbonate soln. in other. The method is suitable for pure soln. tissue extracts and is sensitive to 10 μg of each the compounds named.

J. I. M. Jone

2108. **The use of triethylene glycol in the pa chromatography of steroids.** L. Stárka (Res. I of Endocrin., Prague). *J. Chromatography*, 19 **4** (4), 334–338 (in English).—Paper impregna with $\simeq$30% of triethylene glycol is suitable for separation of a wide variety of steroids.

values of 26 steroids in cyclohexane, light roleum, CCl_4, benzene and toluene are given. peated development, first with CCl_4, then with t petroleum, is recommended for the separation determination of neutral urinary 17-oxosteroids.
R. M. Rowley

109. **Simple method for the determination of um cholesterol.** D. Watson (Royal Women's sp., Melbourne, Australia). *Clin. Chim. Acta*, 0, **5** (5), 637–643.—The method is a modification hat of Pearson *et al.* (*Anal. Chem.*, 1953, **25**, 813). the serum (0·2 ml) add glacial acetic acid 2 ml) then reagent mixture (5 ml), and allow to l (10 to 15 min.). Add conc. H_2SO_4 (0·6 ml), immediately by agitation until the ppt. has solved and read the colour at 560 mμ after ± 2 min. A blank (0·2 ml of H_2O and 0·2 ml of tic acid) and a standard [0·2 ml of H_2O and ml of cholesterol soln. (400 mg per 100 ml in cial acetic acid)] are examined with the test. e reagent mixture is prepared by mixing 3 vol. acetic anhydride with 1 vol. of 0·25M-2,5-ethylbenzenesulphonic acid (**I**) in glacial acetic d and 1 vol. of glacial acetic acid. If the serum haemolysed or jaundiced, a serum blank is essary. For this, add 10-vol. H_2O_2 [prepared by ing 100-vol. H_2O_2 with glacial acetic acid (1 : 9)] 2 ml) to serum (0·2 ml), mix, and add 0·25M-**I** 3 ml). Mix, allow to stand for 20 min. and asure the colour at 560 mμ against **I** soln.
H. F. W. Kirkpatrick

110. **Two improved methods for serum cholesterol imation.** J. Billimoria and D. C. O. James estminster Hosp. Med. Sch., London, England). *n. Chim. Acta*, 1960, **5** (5), 644–651.—In the t method, the cholesterol is extracted from um with acetone - ethanol (1 : 1), the extract is aporated and the Liebermann - Burchard colour ction is carried out on the residue dissolved in cial acetic acid. In the second method, the um is extracted with ethanol - ether (3 : 1), the ract is treated with KOH soln. to hydrolyse ers, the alkali is neutralised and, after evaporation the solvents, the Liebermann - Burchard reaction performed on the residue dissolved in glacial tic acid. H. F. W. Kirkpatrick

111. **Determination of cholesterol and coprosterol faecal lipids.** T. Gerson (Fats Res. Lab., D.S.I.R., llington, New Zealand). *Biochem. J.*, 1960, **77**, 446–448.—The method, which is based on that Zak *et al.* (*Amer. J. Med. Tech.*, 1957, **23**, 283), used for the simultaneous determination of olesterol and coprosterol, both free and total, in cal lipids. A colour reagent is used which con- s of $FeCl_3$ - H_2SO_4 - acetic acid; this gives a oured cholesterol deriv. with an absorption x. at 560 mμ. Measurement is also made at 0 mμ for the orange - red compound formed by prosterol. Other sterols normally present in ces do not materially affect the results, but the erminations are always made on the digitonides ich are pptd. to eliminate other interfering stances. The coeff. of variation are ±2·80% l ±3·41% and the mean recoveries 100·4% and 8% for cholesterol and coprosterol, respectively. e method is suitable for determinations on batches 6 to 8 samples. J. N. Ashley

112. **Comparative experiments for the chemical ermination of the oestrogen content of urine.** Lutzmann and A. Würterle (Univ.-Frauenklinik, rburg/Lahn, Germany). *Clin. Chim. Acta*, 1960, **5** (5), 727–731.—The method of Ittrich (*Anal. Abstr.*, 1959, **6**, 3136) is reliable only when the oestrogens are present in high concn. The method has no significant advantages over that of Brown (*Anal. Abstr.*, 1955, **2**, 3161).
H. F. W. Kirkpatrick

2113. **Effect of oxygen on the determination of 17,21-dihydroxy-20-oxosteroids with tetrazolium salts.** C. A. Johnson, R. King and C. Vickers (Anal. Development Group, Standards Dept., Boots Pure Drug Co. Ltd., Nottingham, England). *Analyst*, 1960, **85**, 714–719.—Atmospheric oxygen affects the intensity and stability of the colour formed when 17,21-dihydroxy-20-oxosteroids react with triphenyltetrazolium chloride. In the method recommended, after re-examination of the influence of other factors, 10 ml of aldehyde-free dehydrated ethanol (**I**) containing 120 to 450 μg of steroid is mixed with 2 ml of 2,3,5-triphenyltetrazolium chloride soln. (0·1 g in 20 ml of **I**) and air is displaced from the flask by oxygen-free nitrogen. Tetramethylammonium hydroxide soln. (4 ml of 25% aq. soln. diluted to 100 ml with **I**) (2 ml) is added and the air is again displaced by nitrogen. The mixture is maintained at 30° in a water bath for 1 hr., the cooled liquid is diluted to 25 ml, and the extinction is measured at 485 mμ against a blank soln. A critical examination is made of the reaction with blue tetrazolium as an alternative method. The greater sensitivity and freedom from interference by O with this reagent are outweighed by the high and variable blank values obtained.
A. O. Jones

2114. **Determination of adrenal cortical α-ketolic steroids by quantitative paper chromatography.** G. W. Kittinger (Cincinnati Jewish Hosp., Ohio, U.S.A.). *J. Lab. Clin. Med.*, 1960, **55** (5), 796–802.—Chromatography of the steroids (2 to 20 μg) is carried out on Whatman 3MM paper by the descending technique at 25° with the upper phase of a mixture of toluene, methanol and water (10 : 7 : 3) after equilibration overnight. The chromatograms are dried in air, treated with an alkaline blue tetrazolium reagent and then scanned in a recording and integrating densitometer. Hydrocortisone, cortisone, corticosterone, aldosterone and other adrenal steroids extracted with dichloromethane from biological fluids may be determined separately, and the method is applied to the *in vitro* secretions of rat adrenal gland. W. H. C. Shaw

2115. **An improved rapid method for free and conjugated 17-hydroxycorticosteroids in urine.** L. Kornel (Univ. Birmingham, England). *Metab., Clin. and Exp.*, 1959, **8**, 432–440.—An improved procedure, based on the Porter - Silber colour reaction, for the detection of free and conjugated 17-hydroxycorticosteroids in urine is described, with the following advantages—the intensity of background colour is half to a third lower; various non-specific substances that give a yellow colour with the phenylhydrazine reagent, *e.g.*, L-ascorbic acid and sugars, are largely eliminated; and almost identical values for conjugated and total 17-hydroxycorticosteroids are obtained with and without the colour correction of Allen. Reproducibility of the method, expressed as the coefficient of variation, is 2·4 and 2·2% for free and conjugated steroids, respectively. Recoveries, by using a concentrated butanol extract of urine, were 96 to 102% and 88 to 100% for free and conjugated corticosteroids. Chem. Abstr.

2116. **Modification of Silber - Porter method for urinary 17-hydroxycorticosteroids conjugated with glucuronic acid.** L. Kornel (Univ. Alabama, Birmingham, U.S.A.). *Metab., Clin. and Exp.*, 1960, **9**, 82–84.—Difficulties encountered in the hydrolysis of 17-hydroxycorticosteroids conjugated with glucuronic acid are overcome by fivefold dilution of the urine with acetate buffer and H_2O after extracting the free 17-hydroxycorticosteroids with $CHCl_3$. The diluted urine is then hydrolysed with β-glucuronidase; dilution of the urine enables a considerable excess of the enzyme to be used. Tests with tetrahydrocortisone monoglucuronide indicated 90 to 100% hydrolysis. The liberated steroids are determined by the method of Kornel (*Ibid.*, 1959, **8**, 432), by which the use of column chromatography and micro-cells is avoided. Reproducibility of the method as applied to dil. urine is 3%, expressed as the coeff. of variation. The 24-hr. urinary output of 17-hydroxycorticosteroid glucuronides estimated in 40 normal subjects was 3 to 14 mg. CHEM. ABSTR.

2117. **Use of α-*N*-(γ-DL-glutamyl)aminonitriles for the colorimetric determination of a specific peptidase in blood serum.** A. Szewczuk and M. Orlowski (Dept. of Biochem., Inst. of Immunol. and Exp. Therapy, Wrocław, Poland). *Clin. Chim. Acta*, 1960, **5** (5), 680–688 (in English).—The method is based on the determination of free α-aminopropionitrile formed with glutamic acid by enzyme hydrolysis of the chosen substrate α-*N*-(γ-DL-glutamyl)aminopropionitrile, synthesis of which is described. The α-aminopropionitrile reacts with Br to give a substance which, in the presence of pyridine and benzidine hydrochloride, produces an intense red colour suitable for quantitative determination. The method is described in detail and results with normal and pathological sera are given.
H. F. W. KIRKPATRICK

2118. **Production and control of a stable penicillinase.** F. W. Bowman and S. Holdowsky (Food and Drug Admin., Washington, D.C., U.S.A.). *Antibiot. & Chemother.*, 1960, **10** (8), 508–514.—A study of the potencies of some commercial penicillinase (**I**) preparations revealed a wide range of activity and indicated the need for more precise control of the **I** required for sterility testing. Details are given of the isolation and maintenance of a stable mutant of *Bacillus cereus* 5/B and for the preparation of high-potency **I**, the stability of which is studied under various conditions. Methods for the assay of **I** are given. W. H. C. SHAW

2119. **Colorimetric method for the determination of isocitric dehydrogenase.** J. L. Bell and D. N. Baron (Royal Free Hosp., London, England). *Clin. Chim. Acta*, 1960, **5** (5), 740–747.—The method depends on the reaction of the α-oxoglutarate, formed by the enzyme action, with 2,4-dinitrophenylhydrazine and alkali to give a coloured phenylhydrazone. Results by this method agree well with those given by spectrophotometric assay of the TPNH formed in the enzyme action.
H. F. W. KIRKPATRICK

2120. **Revised spectrophotometric methods for the determination of glutamic - oxalacetic transaminase, glutamic - pyruvic transaminase and lactic acid dehydrogenase.** R. J. Henry, N. Chiamori, O. J. Golub and S. Berkman (Bio-Science Res. Found., 12318 Santa Monica Boulevard, Los Angeles 25, Calif., U.S.A.). *Amer. J. Clin. Path.*, 1960, **34** (4), 381–398.—More strictly standardised methods the determination of these enzymes are propos
D. E. EVAN

2121. **Determination of 1-phosphofructaldol and sorbitol dehydrogenase. Differential diagn of liver diseases.** W. Brecht and I. Künkele (K Olga Krankenhaus., Stuttgart, Germany). *K Wochschr.*, 1960, **38** (18), 936–938.—1-Phosp fructaldolase (**I**) is determined by the method Wolf *et al.* (*Gastroenterologia*, 1957, **87**, 173), modified for clinical use by Jenny (Inaug. Di Zürich, 1958). This method is based on the sa principles as the more complex optical tests Warburg (*Biochem. Z.*, 1943, **314**, 399). Fructose phosphate is split into dihydroxyacetone phosph (**II**) and D-glyceraldehyde by **I** and, by the addit of α-glycerophosphate dehydrogenase and reduc diphosphoropyridine nucleotide (DPNH), **II** converted into D-glycerophosphate and the DP is oxidised to an equiv. amount of diphospho pyridine nucleotide (DPN). The change in exti tion of the reaction mixture after incubation 10 min. at 36° is measured, and the enzyme activ is given by the change in extinction per min. ml at 36°. Sorbitol dehydrogenase is determined the method of Gerlach (*Klin. Wochschr.*, 1957, 1144; 1959, **37**, 93), in which sorbitol dehydrogen catalyses the reaction—

fructose + DPNH + H^+ → sorbitol + DPN.

The change in extinction due to the formation DPN is used as the index of enzyme activi Absolute specificity of these determinations in li disease is claimed. D. B. PALMER

2122. **Estimation of phospholipase-A activity aqueous systems.** W. L. Magee and R. H. Thompson (Dept. of Chem. Path., Guy's Ho Med. Sch., London, England). *Biochem. J.*, 19 **77** (3), 526–534.—A system containing ether a buffered with 2,4,6-collidine (0·05M, pH 6·5) described, in which the hydrolysis of ovolecit by snake-venom phospholipase-A goes rapidly completion. The enzyme activity is measured the decrease in acyl ester bonds during the reacti and the ester-bond content is determined by hydroxamic acid method. The method is simple a sensitive and requires only small amounts of su strate. This assay system is used for the detect and determination of phospholipase-A activity commercial pancreatin. J. N. ASHLE

See also Abstracts—**1820**, Radioactivation analy in biochemistry. **1862**, Calcium in plant tiss **2000**, Mercapto-groups in biological materi **2006**, Determination of tetroses. **2011**, Detect of ammonium salts of aliphatic acids. **21** Detection of Segontin in serum. **2139**, Fluorine tissues. **2208**, Automatic blood analysis. **22** Apparatus for protein determination. **22** Apparatus for O in blood.

Pharmaceutical analysis

2123. **Elution of alkaloids from citrate-buffe paper chromatograms.** E. G. C. Clarke (Ro Vet. Coll., Royal College Street, London, N.W *Nature*, 1960, **188**, 411.—Treatment of the citra buffered paper with a 10% soln. of $BaCl_2$ conve the citrate into the Ba salt, which is much l soluble in organic solvents. This enables alkaloid spots to be eluted with $CHCl_3$ and col tests to be applied without interference from citr ions. C. B. BAINE

2124. **Colorimetry of ergot alkaloids.** Z. Vejdělek d B. Kakáč (Res. Inst. Pharm. and Biochem., ague). *Českosl. Farm.*, 1960, **9** (7), 359–364.— survey and critical evaluation of methods are esented, with 101 references. J. ZÝKA

2125. **Recommended methods of assay of crude ugs. Assay of rauwolfia.** Joint Committee of e Pharmaceutical Society and the Society for alytical Chemistry on Methods of Assay of Crude rugs. *Analyst*, 1960, **85**, 755–764.—The ground mple is triturated with acetic acid, stirred casionally for 2 hr. and then extracted with hanol for 4 hr. An aliquot of the extract is added 200 ml of dil. H_2SO_4 ($\simeq$0·5N) and is extracted th trichloroethane, each extract being washed th dil. H_2SO_4 and then with $NaHCO_3$ soln. The l. of the filtered extracts is adjusted to 100 ml, plicate aliquots (20 ml) are evaporated to dryness d the residues are dissolved in ethanol and dil. $_2SO_4$. To one soln. (the test soln.) is added 2 ml 0·3% $NaNO_2$ soln., both soln. are heated at 55° r 30 min., then to each is added 5% sulphamic id soln. (1 ml) and the vol. is adjusted to 20 ml th ethanol. The extinction of the test soln. is easured against the blank soln. at 390 mμ and ferred to a calibration graph prepared by treating ln. of reserpine with $NaNO_2$ in the same manner. or the qual. detection of reserpine or rescinnamine, e soln. of alkaloid is evaporated first on a water ath then to dryness at room temp. A soln. of anillin (2 or 3 drops) is added and the mixture is bbed with a glass rod. A pink colour develops ithin 2 min. if reserpine or rescinnamine (down to μg) is present. A. O. JONES

2126. **Separation and determination of some uwolfia alkaloids in mixtures. III. Colorimetric termination of ajmaline, ajmalicine, rescinnamine nd sarpagine.** Z. Jung and H. Petříková (State st. for Control of Drugs, Prague). *Českosl. arm.*, 1960, **9** (8), 385–396.—Reserpine and rescinmine are extracted quantitatively from 0·5N-HCl edium into $CHCl_3$, whereas ajmaline, yohimbine nd sarpagine remain in the aq. phase. All alkaids, except sarpagine (because of its phenolic haracter), can be extracted into $CHCl_3$ from an kaline medium (KOH). Sarpagine can be exacted from an aq. soln. satd. with NaCl and made kaline with aq. NH_3, into a mixture of $CHCl_3$ and opropyl alcohol. Ajmaline and yohimbine, which annot be separated by extraction, can be separated y paper chromatography. Alkaloids separated ccording to the scheme described can be deterined photometrically with xanthhydrol (*Anal. bstr.*, 1958, **5**, 1964) or 1-nitroso-2-naphthol (for rpagine) as reagent. Detailed procedures for the nalysis of pharmaceuticals are given. J. ZÝKA

2127. **Paper chromatography of the tetracyclines.** . G. Kelly and D. A. Buyske (Lederle Lab., Pearl iver, N.Y., U.S.A.). *Antibiot. & Chemother.*, 960, **10** (10), 604–607.—Chromatography is carried ut on Whatman No. 1 paper treated with 0·1M-DTA (disodium salt) and then dried. Descending evelopment is for 16 to 20 hr. with n-butanol - aq. H_3 - water (4:1:5) in an atmosphere of N or ith n-butanol - acetic acid - water (4:1:5). The ried chromatograms are exposed to NH_3 vapour nd examined under u.v. light. R_F values for 12 tracyclines in both systems are given, and the use the method in metabolic studies is discussed. W. H. C. SHAW

2128. **Improved assay for psicofuranine.** L. S. Hanka and M. R. Burch (Upjohn Co., Kalamazoo, Mich., U.S.A.). *Antibiot. & Chemother.*, 1960, **10** (8), 484–487.—Unsatisfactory results by the disc-plate microbiological assay described previously (*cf. Anal. Abstr.*, 1960, **7**, 1896) are attributed to the presence of an inhibitory substance in the liver extract included in the medium. The synthetic medium recommended gives satisfactory responses with *Staphylococcus aureus* FDA-209P over the range 10 to 80 μg of psicofuranine per ml. W. H. C. SHAW

2129. **Determination of iodoform by photo-oxidation in artificial light.** R. M. Verma and Sameer Bose (Mahakoshal Mahavidyalaya, Jabalpur, India). *J. Indian Chem. Soc.*, 1960, **37** (9), 540–542 (in English).—The method is essentially that of Bose (*cf. Anal. Abstr.*, 1958, **5**, 4299), with a mercury vapour lamp as the source of illumination instead of sunlight. W. T. CARTER

2130. **Colorimetric determination of phenacetin in tablet mixtures.** K.-T. Lee and C.-S. Chan (Gov. Dept. of Chemistry, Outram Road, Singapore). *J. Pharm. Pharmacol.*, 1960, **12** (10), 624–630.—Phenacetin (**I**) is quant. nitrated and saponified and the nitrophenetidine produced is determined spectrophotometrically. *Procedure*—Evaporate to dryness a soln. containing **I** (1 to 5 mg) in $CHCl_3$, add 50% (v/v) HNO_3 (2 ml) and set aside for 30 min. at room temp.; add 10% NaOH soln. (10 ml), heat on a steam bath for 30 min., dilute with H_2O to 100 ml and measure the extinction at 445 mμ. Caffeine (**II**) does not interfere. To determine **I**, **II** and aspirin (**III**) in commercial tablets, shake the powdered tablets (0·5 g) with H_2O (10 ml), 6% $NaHCO_3$ soln. (15 ml) and dil. aq. HCl (2 drops), extract with $CHCl_3$ (5 × 30 ml) and pass each $CHCl_3$ portion through the same 3 ml of 6% $NaHCO_3$ soln. Acidify the combined $NaHCO_3$ soln. with aq. HCl and extract and determine **III** by the A.O.A.C. method ("Methods of Analysis," 8th Ed., p. 619). Determine **I** in a 5-ml aliquot of the combined $CHCl_3$ soln. by the method given above. Determine **II** in a 10- or 15-ml aliquot of the $CHCl_3$ soln. by the method of Daoust (*cf. Anal. Abstr.*, 1954, **1**, 1058). A. R. ROGERS

2131. **Spectrophotometric determination of salicylamide, *p*-aminobenzoic acid, phenacetin and caffeine in pharmaceuticals.** W. Żyżyński (Drug Res. Inst., Warsaw). *Acta Polon. Pharm.*, 1960, **17** (4), 277–286.—The absorption curves for each of these compounds were prepared and their use in analysing certain mixtures was investigated. A mixture of salicylamide (**I**) and *p*-aminobenzoic acid is determined at 265 mμ and 326 mμ; a mixture of **I** and caffeine at 272 mμ and 326 mμ; and a mixture of **I**, caffeine and phenacetin at 244 mμ, 272 mμ and 326 mμ. The sample is dissolved in ethanol and buffered to pH 10, and the components are determined spectrophotometrically against a blank (ethanol in buffer soln.) without previous separation. B. K.

2132. **Detection of *N*-(1-methyl-2-phenylethyl)-3,3-diphenylpropylamine.** A. Häussler (Pharm.-Wissenschaftl. Abt., Farbwerke Hoechst A.-G., Frankfurt a. M.-Höchst). *Arzneimittel-Forsch.*, 1960, **10** (8), 585–588.—*N*-(1-Methyl-2-phenylethyl)-3,3-diphenylpropylamine (Segontin) (**I**) lactate is identified as its reineckate, m.p. 181° (uncorr.) and by formation of cryst. ppt. with $AuCl_3$ and $PtCl_4$.

To determine **I**, add 1 ml of 2N-HCl to 5 ml of aq. soln. containing 100 to 2000 μg of **I**, extract into 5 ml of $CHCl_3$ and measure the extinction at 259 mμ. The method is unsuitable for the determination of **I** in biological fluids. Alternatively, mix 5 ml of soln. (containing 30 to 180 mg of **I** per 100 ml) and 3 ml of cold, saturated reineckate soln., filter, wash the ppt. with very dil. reineckate soln., dry at 90° for 1 hr., dissolve in acetone and read the extinction of the soln. at 545 mμ. To determine **I** in urine or serum, Tropaeolin OO (**II**) is used. Mix 5 ml of soln. containing 20 to 200 μg of **I**, 5 ml of acetate buffer (pH 4·62) and 3 ml of 0·1% **II** soln., extract with $CHCl_3$ (4 × 5 ml), add methanolic H_2SO_4 [1% (v/v)], dilute to 50 ml with $CHCl_3$ and measure the extinction at 545 mμ. To determine **I** in serum, first precipitate the albumin by addition of one-third vol. of ethanol and proceed as described above.

A. G. Cooper

2133. **Quantitative fluorimetric reaction for glutethimide.** R. P. Haycock, P. B. Sheth and W. J. Mader (Res. Dept., Ciba Pharmaceutical Products Inc., Summit, N.J., U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (10), 673–677.—Heat a soln. of glutethimide (**I**) (≃0·5 mg) in methanol (1 ml) with a freshly prepared 3·7% soln. of formaldehyde in conc. H_2SO_4 (5 ml) for 20 min. at 100° in an oven, cool to room temp., dilute to 50 ml with 50% H_2SO_4, shake well and compare the intensity of fluorescence at 450 mμ excited by radiation of 365 mμ with readings of a standard and a reagent blank. Common tablet excipients, such as starch, sucrose, lactose, talc and stearates, do not interfere. The main degradation product of **I**, 4-ethyl-4-phenylglutaramic acid, yields only one-tenth of the fluorescence of **I**. A. R. Rogers

2134. **Spectrophotometric method for the assay of glycobiarsol [bismuth glycollylarsanilate].** M. E. Auerbach and W. W. Houghtaling (Sterling-Winthrop Res. Inst., Rensselaer, N.Y., U.S.A.). *Drug Standards*, 1960, **28** (5), 115–116.—Shake the finely powdered tablets (containing ≃125 mg of glycobiarsol) for 5 min. with 5% aq. EDTA (disodium salt) soln. (25 ml), dilute with H_2O to 250 ml, mix and filter through paper. Mix 2 ml of the filtrate with N-HCl (1 ml), dilute with H_2O to 100 ml and measure the extinction at 258 mμ. The results are in close agreement with those obtained by the method of the [U.S.] N. F. X, and are obtained more rapidly and easily. A. R. Rogers

2135. **Selective determination of isopropamide iodide, a low-molecular-weight quaternary ammonium compound.** R. S. Santoro (Smith Kline and French Laboratories, Philadelphia, Pa., U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (10), 666–668.—*Procedure*—Mix a soln. of isopropamide iodide [(3-carbamoyl-3,3-diphenylpropyl)methyldiisopropylammonium iodide] (**I**) (0·05 mg) in 0·1N-HCl (5 ml) with methyl orange buffer soln. [shake methyl orange (2 g) with phosphate buffer soln. of pH 10·2 (1 litre), filter and extract with $CHCl_3$ until the $CHCl_3$ layer is colourless] (20 ml) and extract with $CHCl_3$ (4 × 25 ml). Filter the extracts through cotton wool and wash the cotton wool with $CHCl_3$ (25 ml). Shake the combined filtrate and washings with N-HCl (10 ml), allow the layers to separate and measure the extinction of the aq. phase in a Klett - Summerson photometer with a No. 52 filter. Subtract a reagent blank. Other short-chain quaternary amines down to the tetra-n-propyl compound may be determined similarly. Primary, secondary and tertiary amines and alka loids do not interfere. The method has been applie to the determination of **I** in the presence of pr chlorperazine dimaleate in a pharmaceutical pr paration; recoveries in 5 assays ranged from 98 to 101·3%. A. R. Rogers

2136. **Studies of anticoagulants. XXXIII. Dete tion reactions for paper chromatography of coumari and related substances.** Z. Ledvinová and I. M Hais (Res. Inst. Pharm. and Biochem., Prague *Československ. Farm.*, 1960, **9** (8), 401–409.—The detectio of 77 compounds of the coumarin and chromon type is described, with the use of the origina fluorescence of the compounds, of the fluorescenc after treatment with NaOH (Fučík and Kořistek *Chem. Listy*, 1952, **46**, 190), coupling with diazotise 4-nitroaniline or tetrazotised benzidine, and th reactions with Dragendorff and Ehrlich reagents.

J. Zýka

2137. **Polarographic determination of 2,4-diamino 4′-ethoxyazobenzene (Urokarmin).** L. Fait (State Inst. for Contr. of Drugs, Bratislava Czechoslovakia). *Farmácia*, 1960, **29** (9), 286–288.— 2,4-Diamino-4′-ethoxyazobenzene (**I**) is polaro graphically reduced in soln. of pH 2 to 6, th best developed two-electron wave being produced i pH 4·2 soln. ($E_{\frac{1}{2}}$ = −0·30 *vs.* the S.C.E.). *Pro cedure*—Dissolve the sample containing abou 0·05 g of **I** by gently heating in ethanol (96% (40 ml), cool and dilute to 50 ml. Mix 0·50 ml o this soln. with acetate buffer soln. (pH 4·2), dilut to 10 ml, remove O with a stream of N, register th wave and compare with a calibration curve. Thi procedure, which yields results with an error o ±3%, can be used for the determination of **I** i tablets. J. Zýka

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See also Abstracts—**1819**, Gas chromatography i anaesthetic research. Stability of medicinal paraf fin. **2000**, Mercapto-groups in drugs. **2014** Analysis of nitric acid esters. **2044**, Cineole i eucalyptus oil. **2113**, **2114**, Determination o corticosteroids. **2118**, Potency of penicillinase.

Food

Foods and food additives, beverages, edible oils and fats, vitamins.

2138. **Procedure for the determination of organi acids in foods.** J. Schormüller and H. Langne (Inst. f. Lebensmittelchem. u.- technol., Techn Univ., Berlin). *Z. Lebensmitt Untersuch.*, 1960, **11** (2), 104–112.—Full directions are given of pro cedures for the qual. and quant. analysis of mixture of (*i*) volatile fatty acids (C_2 to C_9), (*ii*) non-volatil aliphatic mono-, di- and tri-carboxylic acids (eight and (*iii*) α-oxocarboxylic acids (four). The qual analyses are carried out by circular paper chro matography of the ammonium salts, free acids, an the dinitrophenylhydrazones, respectively, of th three groups. The acid mixtures obtained b steam-distillation are analysed quant. by ga chromatography by the method of James an Martin (*Biochem. J.*, 1951, **50**, 679). Mixtures o group (*ii*) are quant. analysed by the gradient elu tion technique of Busch *et al.* (*J. Biol. Chem.*, 1952 **196**, 717) supplemented by the colorimetric deter mination of lactic acid by the method of Barke and Summerson (*Ibid*, 1941, **138**, 535), and by th gradient elution of the fraction containing tartaric

ionic and citric acids from a column of silicic l with mixtures of butanol and $CHCl_3$ (1:2 and). Mixtures of group (*ii*) are separated by the tion of the dinitrophenylhydrazones from a mn of Celite 545 by ether containing, progres- ly, 2, 3 and 5% of ethanol, and quant. determined spectrophotometric measurement at 450 mμ. uvic and α-oxobutyric acids cannot be separated this method, but can readily be distinguished by chromatographic method. From mixtures of own composition, 97% of the acids of groups (*i*) (*ii*), and 92% of those of group (*iii*) can be overed, with the exception of *cis*-aconitic and lylacetic acids, for which the recoveries are 70 89%, respectively. P. S. Arup

2139. **The determination of fluorine in animal and etable tissues and in foods.** E. Matthey, F. Fassa V. Demole (Lab. Cantonal, Lausanne, Switzer- d). *Mitt. Lebensmitt. Hyg., Bern*, 1960, **51** (5), 339– (in French).—The micro-method of Banerjee *nal. Abstr.*, 1956, **3**, 1350) was tested and used for determination of fluorine in urine, blood, bones, th and various foods. The method was found be reliable. A method of purification of $Ca(OH)_2$ m F is given and the apparatus used is described. I. Dickinson

2140. **Extraction of antimony from enamelled ensils into food.** H. Mazur (State Inst. of Hygiene, arsaw). *Roczn. Zakł. Hig., Warsaw*, 1960, **11** (3), 7–225.—A sample of food, after being cooked in vessel, is dry-ashed, the ash is dissolved in 10% SO_4, and KI soln. and thiourea are added. The ensity of the yellow colour is measured in a lfrich photometer (No. 8 filter) and compared th a calibration curve, obtained by the same ocedure from soln. of metallic antimony in H_2SO_4. e method gives good results for amounts of $_2O_3$ from 0·1 to 0·5 mg. The content of Sb in e kinds of food, cooked in 5 kinds of utensil, was termined; 3% soln. of tartaric, acetic, lactic and ric acids were also investigated by the same ethod. The amounts of Sb obtained were much wer than the toxic dose. B. K.

2141. **Determination of sugars in chocolate ntaining glucose.** M. Borghi. *Boll. Lab. Chim. ov.*, 1960, **11** (3), 376–384.—Sucrose is determined larimetrically before and after enzymatic inver- on. In sucrose - lactose or sucrose - glucose ixtures, lactose or glucose is determined in an iquot by the Luff - Schoorl method. Alternatively, e glucose can be determined by the Auerbach - odländer iodimetric method (*Ind. Sacc. Ital.*, 1935, , 4). When all three sugars are present an iquot is fermented with yeast, which removes crose and glucose, leaving lactose to be deter- ined by the Luff - Schoorl method. J. I. M. Jones

2142. **Analysis of milk chocolate.** M. Borghi. *oll. Lab. Chim. Prov.*, 1960, **11** (3), 368–375.— ne identification of whole-milk powder is based on e ratio of milk fat to lactose, which, for whole ilk, is taken as 0·66. The analysis for total milk mponents in milk chocolate includes the deter- ination of milk fat, lactose and milk proteins. *Milk fat*—The total fat and the semi-micro butyric id index (Grossfeld, *Z. Untersuch. Lebensmitt.*, 938, **76**, 340) are determined. Milk fat is given y total fat × B.I./20, where B.I. is the butyric id index. *Lactose*—If lactose is the only reducing gar present it is determined by the Luff - Schoorl method. If other sugars are present a biochemical method must be used (*cf. Rev. Int. Chocolat.*, 1954, **9**, 3). Paper chromatography enables the presence of glucose, fructose and maltose to be established by Thaler's method (*Rev. Int. Chocolat.*, 1958, **13**, 2; *cf. Anal. Abstr.*, 1959, **6**, 1095). *Milk proteins*— Extract 10 g of finely grated chocolate in a centrifuge tube with ethyl ether (2 × 100 ml). Remove the ether, shake the residue well with water (100 ml) for 4 min. then add 1% Na oxalate soln. (100 ml). Shake and centrifuge for 15 min. at 1800 r.p.m. To the supernatant liquid (100 ml) add 4N-acetic acid (1 ml). Allow the ppt. to settle and add, little by little with stirring, fresh 10% tannin soln. (4 ml). Filter through S. & S. paper 589 covered with a layer of Whatman paper-pulp. Wash with 0·4N-acetic acid - 10% tannin soln - 1% Na oxalate soln. (1:2:1) two or three times. Transfer the filter and ppt. to a Kjeldahl flask and determine the N; protein (%) = mg of N × 6·38 × 2F, where F is an empirical factor (= 1·05). The total milk components are the sum of the milk fat, protein, lactose and salts, which are taken to be 20% of the lactose. J. I. M. Jones

2143. **Potentiometric determination of chloride in milk.** B. L. Herrington and D. H. Kleyn (Cornell Univ., Ithaca, N.Y., U.S.A.). *J. Dairy Sci.*, 1960, **43** (8), 1050–1057.—Interference of protein in the potentiometric determination of Cl^- in milk is eliminated by adjusting the pH to 2 with aq. HNO_3 before titration with $AgNO_3$. *Procedure*—To 10 ml of the sample in a beaker add 20 ml of aq. HNO_3 (6 ml of conc. HNO_3 per litre) and titrate (stirring) with aq. $AgNO_3$ (4·882 g per litre), with a silver electrode to a potential of +250 mV *vs.* a S.C.E. Use of a S.C.E. with an asbestos filter-tip prevents diffusion of Cl^- during titration. Potassium aluminium sulphate may also be used in place of aq. HNO_3 for adjusting the pH. W. H. C. Shaw

2144. **Simplified method for the determination of iodine-131 in milk.** G. K. Murthy and J. E. Campbell (R. A. Taft, Sanitary Engng Center, Cincinnati, Ohio, U.S.A.). *J. Dairy Sci.*, 1960, **43** (8), 1042–1049.—The method depends on the conversion of inorganic ^{131}I in milk into protein-bound ^{131}I when the sample, treated with formaldehyde, is allowed to stand for 1 to 2 hr. The proteins are then pptd. with trichloroacetic acid, separated and submitted to a γ-radiation counting technique. Recovery of ^{131}I is 98 (±2) % and the presence of other γ-emitters (^{40}K, ^{137}Cs and ^{140}Ba plus ^{140}La) has no effect on the results. W. H. C. Shaw

2145. **Improved procedure for the determination of milk proteins by dye binding.** U. S. Ashworth, R. Seals and R. E. Erb (Washington State Univ., Pullman, U.S.A.). *J. Dairy Sci.*, 1960, **43** (5), 614–623.—The dye-binding method with Orange G (C.I. Acid Orange 10) is modified by the use of a special flow-through cell. Improved precision and good agreement with total protein (N × 6·38) values are attained, and the results are independent of fat concn. Calibration, precision, the dye-binding capacity of proteins and non-protein matter, and the use of sample preservatives are discussed. W. H. C. Shaw

2146. **Rapid determination of protein in fresh milk.** F. Kiermeier and E. Renner (Milchwirtschaftl. Inst. d. Tech. Hochsch., München in Weihenstephan, Germany). *Z. LebensmittUntersuch.*, 1960

113 (1), 1–13.—The literature is reviewed. Results from 51 samples obtained by formol titration by the Schulz procedure (*MolkereiZtg*, 1952, **3**, 1412) with the use of K oxalate and the Kofranyi method (*Milchwissenschaft*, 1950, **5**, 51), and by two colorimetric methods with Amido black (C.I. Acid Black 1) [Steinsholt (*Meiereiposten*, 1957, **46**, 259, 901) and Schober and Hetzel (*Anal Abstr.*, 1957, **4**, 4121)] all agree satisfactorily with the corresponding Kjeldahl results. The formol titration method is preferred on account of its comparative rapidity; minor modifications are proposed in order to increase the accuracy and convenience of the Schulz procedure. Calibration graphs are given for the two colorimetric methods. The increase in the formol titration and Kofranyi results frequently observed during the souring of milk is due, not to the action of lactic acid bacteria, but to the proteolytic activity of *Bacillus macerans*, which thrives in an acid medium. (73 references.) P. S. Arup

2147. **Routine method for determining sucrose in sweetened condensed milk.** F. E. Murphy (United Dairies Res. Lab., Wood Lane, London). *Analyst*, 1960, **85**, 720–723.—The sample ($4 \pm 0{\cdot}005$ g) is mixed with hot water (> 25 ml) and 0·5 ml of 10% w/v citric acid soln. and diluted to 50 ml at 20°. The liquid is filtered (soln. *A*) with rejection of an initial portion. A subsequent 50-ml portion is mixed with 20 ml of water and 30 ml of the citric acid soln. and gently boiled for 10 min., avoiding concentration. The liquid is rapidly cooled (> 30°) and 5N-NaOH is run in until a piece of litmus paper previously added just changes colour. The liquid is then diluted to 250 ml (soln. *B*). The reducing power of this soln. is determined by Lane and Eynon's method, 23 ml being added to 10 ml of Fehling's soln. as the first incremental addition for full-cream milk and 21 ml for separated milk. The reducing power of soln. *A* is determined by adding 25 ml for a full-cream milk and 20 ml for a separated milk and 5 ml of Calgon soln. (10%) to 10 ml of Fehling's soln., the incremental titration being continued with soln. *B*. The method of calculation is given.
A. O. Jones

2148. **Chromatographic analysis of fatty substances in the vapour phase. III. Preparation of chromatograms of butter and related products.** S. Anselmi, L. Boniforti and R. Monacelli (Ist. Super. Sanità, Roma, Italy). *Boll. Lab. Chim. Prov.*, 1960, **11** (3), 317–330.—Gas-chromatographic analysis of acids in butter and related products is carried out on the ethyl esters of the fatty acids, the methyl esters being too volatile and soluble in water to give satisfactory results. A single chromatogram of the completely esterified fat gives unsatisfactory results for fatty acids below C_{10}. Two chromatograms are therefore made, one on the total acids, which gives qual. separation of acids above C_{10}, and another on a vacuum-distilled fraction of the esters, which gives a similar separation of those up to C_{10}. Quantitative separation is the subject of further study.
J. I. M. Jones

2149. **Quantitative determination of carbohydrates in ice-cream by paper chromatography.** T. C. Chou and J. Tobias (Illinois Univ., Urbana, U.S.A.). *J. Dairy Sci.*, 1960, **43** (8), 1031–1041.—A 30% w/v aq. dilution of the melted sample is spotted directly on to Whatman No. 1 paper for development by the descending technique with n-butanol - ethanol - water (2:1:1). Equilibration is for 6 24 hr. and development for 100 to 115 hr. App priate areas (located by guide strips, cut out a sprayed with aniline phthalate) are cut out a eluted with 25 ml of water. The solutions filtered and the sugars in aliquots are determi with an anthrone reagent. The recovery is 93 104%, averaging 100%. W. H. C. Shaw

2150. **Rapid method for the determination sucrose in ice-cream.** L. Reinhardt (Morgenleite Dresden A 36). *Nahrung*, 1960, **4** (5–6), 533–536. The use of tables for the calculation of sucrose fr the difference between the determinations bef and after inversion produces a large error wh lactose is present. In the method described, t proteins are precipitated with ethanol, the reduci sugars are destroyed by heating with $Ba(OH)_2$ 70° to 80° and the sucrose is determined pola metrically. I. Dickinson

2151. **Determination of organic fluorine residu in blackcurrants.** H. Egan and R. Wood (D.S.I. Lab. of the Gov. Chemist, Clement's Inn Passag Strand, London). *J. Sci. Food Agric.*, 1960, **2** (1 582–584.—A method, less time consuming than th previously proposed (*cf.* Thompson, *Anal. Abst* 1960, **7**, 1972), is described. It consists in a extraction of the plant material, ether extracti of the fluoroacetic acid from the aq. extract, remov of inorganic fluorine on a silicic acid column, ignitio of the organic fluorine compounds with lime, a distillation of the released fluoride, followed colorimetric determination of the fluoride ion. T duration of the ether extraction was reduced to 8 h without loss of accuracy; two short ignitions 20 min. each gave satisfactory recoveries with pu fluoroacetic acid. A single fluorine distillation a high temp. was found to introduce no interferen in the final colorimetric determination. The colo metric procedure based on bleaching by the fluori ion of the coloured complex of zirconium wi Eriochrome cyanine R (C.I. Mordant Blue 3) w used, because the reaction is rapid.
I. Dickinson

2152. **Rapid determination of DDT and Gam mexane [hexachlorocyclohexane] in flour and grai** G. Paulig. *Dtsch. LebensmittRdsch.*, 1960, **56** (8 223–224.—The method involves extraction wit n-hexane, purification of the extract by chromat graphy and washing with conc. H_2SO_4, and th measurement of the i.r. spectrum of a soln. in CS DDT is determined from its typical double ban at 12·8 to 13 μ, and hexachlorocyclohexane fro its band at 14·6 μ. *Procedure*—Stir the groun sample (100 g) for 30 min. with n-hexane (200 ml and filter (G4 sintered glass). Dry the extract wit Na_2SO_4 and evaporate it to 10 ml under reduce pressure. Transfer the soln. to a 10-cm column c activated alumina and elute with n-hexane (150 ml Reduce the volume to 50 ml under reduced pressur and shake with conc. H_2SO_4 (2 × 50 ml), was twice with satd. aq. $NaHCO_3$ and twice with wate Dry the soln. with Na_2SO_4, evaporate to drynes under reduced pressure, dissolve the residue in CS (5 ml) and measure the extinction in an i.r. spectro photometer. E. C. Apling

2153. **Spectrophotometric determination of sorbi acid in butter and margarine.** L. Olivari and F Benassi (Lab. Chim. Prov., Reggio Emilia, Italy) *Boll. Lab. Chim. Prov.*, 1960, **11** (3), 343–347.—T

sample (5 g) add $MgSO_4.7H_2O$ (50 g), citric acid) and water (50 ml). Steam-distil, at a temp. 30°. Collect the distillate in 10N-NaOH (1 ml). porate an aliquot of the distillate to dryness and olve the residue in H_2O. Filter, if necessary, ough S. & S. paper 589/3 and discard the first nl of filtrate. Dilute 20 ml of the remainder to ml and read the extinction at 255 mμ against a nk prepared in the same way. Prepare a ndard curve with pure sorbic acid. The re- ery is 96%. The error due to other substances racted from butter and margarine is small and is wed for by deducting 0·003 to 0·005 from the ctrophotometric reading. The method is applic- e to slightly rancid materials. J. I. M. Jones

154. **Analysis of piperine by spectrophotometric hod. I.** T. N. Ramachandra Rao, C. T. arakanath and D. S. Johar (Cent. Food Tech. . Inst., Mysore). *J. Instn Chem., India*, 1960, (3), 125–129.—The piperine content of the ce and oleoresin from ten varieties and four mmercial grades of pepper has been determined ectly by a spectrophotometric method and by standard A.O.A.C. determination for N. The her results from the latter were attributed to presence of other nitrogenous substances. ttandan oleoresin showed the highest piperine tent (55·12%), with 7·39% in the original ce, while stems and stalks and unfertilised buds tained <1%. Little difference was found ween white and black commercial peppers.

P. M. Kingston

155. **Quantitative determination of volatile oils hops.** H. Bausch, E. Rothenbach and H. Wolter st. f. Gärungschem. u. -technol., Humboldt iv., Berlin). *Nahrung*, 1960, **4** (7), 599–617.— e yield obtained by steam-distillation is normally med "the volatile oil content" of the plant terial. The effects on this yield of such factors the method of distillation, time, speed, prepara- n of sample, whether the steam produces volatile bstances and whether part of the volatile oils dergoes chemical changes during the distillation investigated. Results are given. The distillation standardised, and the method and apparatus are cribed. The distillate is received in 10 ml of ntane which was found to absorb 98% of the ld. The finely dispersed residue is determined ohelometrically. The reproducibility is ±0·02%. e analysis takes 2·5 hr., which makes the method table for routine examinations.

I. Dickinson

156. **Chromatographic micro-identification of rose in wine.** F. de Francesco and D. Avancini ab. Chim., Stazione Sper. Agr., S. Michele all'Adige, nto, Italy). *Boll. Lab. Chim. Prov.*, 1960, **11** , 385–391.—Decolorise the wine (15 to 20 ml) h animal charcoal and filter. To 1 ml of filtrate l an equal vol. of Merck's ion-exchange mixture Shake till the soln. is neutral, then place 20 cu. n on Whatman No. 1 paper and subject to scending chromatography with n-butanol - acetic d - water (8:1:5) for 20 hr. till the solvent front is ar the lower edge of the paper (40 to 45 cm travel). y the paper in a current of air for 30 min. Spray h one of the soln. given below, dry in air and heat 140° for 15 min. The various sugars appear as wn spots on a red ground. Lactose, sucrose, cose and fructose can thus be separated and ntified, the R_F values being respectively 0·05, 9, 0·16 and 0·21; 0·25 g per litre of sucrose can be detected in wine and 0·5 g per litre in must. *Colour-developing soln.*—*A*: Equal volumes of (*i*) aniline (0·93 g) and phthalic acid (1·66 g) in 100 ml of water satd. with n-butanol, (*ii*) benzidine (2 g) in 50 ml of ethanol, and (*iii*) urea (5 g) in 20 ml of HCl and 80 ml of ethanol; *B*: *p*-anisidine (1·23 g) and phthalic acid (1·66 g) in 100 ml of ethanol.

J. I. M. Jones

2157. **Detection of sucrose in dessert wines by paper electrophoresis.** H. Konrad (Inst. f. Lebensmittelchemie beim Magistrat v. Gross-Berlin). *Nahrung*, 1960, **4** (5–6), 528–532.—A rapid method suitable for routine control is described. *Procedure* —The sample (0·02 ml) is applied at the centre of a paper strip 4 cm wide, which is then saturated with borate buffer soln. (0·2M, pH 7·7) and submitted to electrophoresis at 10 to 12 V per cm for 4 hr.; the paper is then dried and sprayed with a mixture of 10 ml of a 2% soln. of 1,3-dihydroxynaphthalene in acetone and 1 ml of 3N-phosphoric acid. A few minutes after drying the sucrose appears as a reddish-brown band, glucose as a pale-blue one and fructose as a brown one. I. Dickinson

2158. **New fluorimetric determination of glycerol in wine analysis.** J. Eisenbrand and M. Raisch (Chem. Untersuchungsamt, Saarbrücken, Germany). *Dtsch. LebensmittRdsch.*, 1960, **56** (9), 257–260.— The method is based on the formation of quinoline by Skraup's synthesis and the measurement by means of a photomultiplier of its blue fluorescence at 420 mμ excited by monochromatic u.v. light of 313 mμ. *Procedure*—Dilute the wine sample with water (9 vol.) and mix this soln. (1 ml) with conc. H_2SO_4 (1·1 ml) and 0·25 g of a mixture of aniline sulphate (20 g) and Na *m*-nitrobenzenesulphonate (5 g) in a tube fitted with an air-condenser. Heat in an oil bath for 30 min. at 150° to 160°, cool and transfer to a steam-distillation apparatus with a satd. soln. of Na_2SO_4 (25 ml). Add 70% KOH soln. (10 ml) and steam-distil, collecting the distillate beneath N-H_2SO_4 (10 ml) in a 100-ml flask. Dilute the distillate with 0·1N-H_2SO_4 to contain between 0·1 and 1 μg of quinoline per ml and measure the intensity of the fluorescence excited by light of 313 mμ. The method is suitable for serial analysis and gives results in good agreement with the periodate method of Rebelein. E. C. Apling

2159. **Chromatographic investigations on dehydracetic acid. Application to wines.** I. Bastianutti and B. Romani (Lab. Chim. Prov. Udine, Italy). *Boll. Lab. Chim. Prov.*, 1960, **11** (3), 331–334.— Extract the wine (100 ml) with ethyl ether (3 × 50 ml). Wash the extract three times with water (100 ml in all) then allow the ether to evaporate at room temp. Treat the residue with 2N-H_2SO_4 (2 ml) and add dropwise 0·1N-$KMnO_4$ till a faint pink colour persists for a few minutes. It is not necessary to dissolve crystals of dehydracetic acid which separate. Extract the residue in a separating-funnel 3 times with ether (100 ml in all), filter, dry with anhyd. sodium sulphate and allow the ether to evaporate at room temp. Dissolve the residue in ether (0·3 ml) and transfer to Whatman No. 1 paper. Subject to ascending chromatography with cyclohexane - dioxan - acetic acid - water (5:4:2:2). After 3 hr. the front will have moved 12 to 15 cm. Dry the chromatogram in air then spray with 0·37% ethanolic $FeCl_3$ soln. Dehydracetic acid gives an orange - yellow spot with $R_F = 0·93$.

J. I. M. Jones

2160. **Chromatographic detection of salicylic and dehydracetic acids in wine.** B. Romani and I. Bastianutti (Lab. Chim. Prov., Udine, Italy). *Boll. Lab. Chim. Prov.*, 1960, **11** (3), 348–350.—Salicylic and dehydracetic acids present simultaneously in wine can be separated and identified by the procedure previously described (*cf. Anal. Abstr.*, 1961, **8**, 2159) with pyridine - 1·5N-aq. NH_3 - water (1:2:2) as solvent. The R_F values are, for dehydracetic acid, 0·57, and for salicylic acid, 0·71.
J. I. M. Jones

2161. **Chromatographic identification of some preservatives in wine.** I. Bastianutti and B. Romani (Lab. Chim. Prov., Udine, Italy). *Boll. Lab. Chim. Prov.*, 1960, **11** (3), 362–367.—To the wine (100 ml) add salicylic acid (1 mg) as a marker and extract three times with ethyl ether (100 ml in all). If it is desired to test for salicylic acid previously present, a second sample is similarly extracted omitting the addition of salicylic acid. Allow the extracts to evaporate at room temp. and treat the residues with 36% H_2O_2 (5 ml) and conc. aq. NH_3 (sp. gr. 0·925) (2·5 ml). Set aside for 4 hr., stirring occasionally; the mixture, originally brown, becomes completely decolorised. Acidify with dil. H_2SO_4 and re-extract with ether as described above. Dry the extract with anhyd. Na_2SO_4 for 20 hr., decant the ether and allow it to evaporate at room temp. Dissolve the residue in ether (1 ml) and transfer to Whatman No. 1 paper for chromatography. Dissolve any insol. matter in methanol and transfer to the ether spot on the paper. Run an ascending chromatogram with butanol - 25% aq. NH_3 - water (7:2:1) for 16 hr. Air-dry the chromatogram and locate the salicylic acid marker in u.v. light. Spray with 1% ethanolic quinine and examine in u.v. light to identify chloro-, bromo- and iodo-acetic acids, benzoic acid and derivatives of *p*-hydroxybenzoic acid as dark spots. Spray that part of the chromatogram below the salicylic acid spot with 0·25% ninhydrin in acetone and dry at 150° for 10 min. Spots due to the amino-acid derivatives formed from halogen-acetic acids and α- and β-bromopropionic acids will assume a blue - violet colour. Spray that part above the salicylic acid spot with Millon's reagent and dry in warm air. Spots due to esters of *p*-hydroxybenzoic acid will appear wine-red. No method for identifying sorbic acid has been devised. J. I. M. Jones

2162. **Identification and determination of hydrocyanic acid in wines treated with ferrocyanide.** P. Jaulmes and R. Mestres (Fac. de Pharm., Montpellier, France). *Ann. Falsif.*, 1960, **53**, 455–475.—The extracted HCN is determined with Epstein's reagent [bi-4-(3-methyl-1-phenyl-2-pyrazolin-5-onyl)] (preparation described). The extinction is measured at 625 mμ. N. E.

2163. **Determination of the methanol content of spirits with chromotropic acid.** L. Uino and T. Salo (Res. Lab., State Alcohol Monopoly, Helsinki, Finland). *Z. LebensmittUntersuch.*, 1960, **113** (2), 129–134.—The effects of variations in the experimental details of the Bremanis procedure (*Ibid.*, 1951, **93**, 1) are examined. The following modifications are embodied in a revised set of directions. The best results are obtained by diluting the sample to a content of 5% of ethanol. The time of oxidation of the diluted sample with $KMnO_4$ - H_3PO_4 soln. may vary from 5 to 30 min. without effect. The optimum concn. of the chromotropic acid reagent is 1·5%; this reagent must be freshly prepared for use; the optimum conditions for colour developm (in the presence of 81% of H_2SO_4) are 30 min. 100°. Spectrophotometric measurements are b made at 575 mμ. If the colour is to be measu >2 hr. after the beginning of the heating, the so must be kept in the dark, but the measurem must be made within 2 days. Extinction grap are given for 1- and 3-cm cells. P. S. Aru

2164. **Ultra-violet spectrophotometric method characterisation of expressed olive oil.** F. Albon (Ist. Ind. Agrarie, Milan Univ., Italy). *Olii Mi* 1960, **37** (8), 343–345.—The maximum limit conjugated triene suggested for super-refined virg olive oil is 1·32 mg per 100 g of oil, corresponding a $\triangle K$ val. of 0·01, where $\triangle K = K_{268} - 0{\cdot}5(K_{262} K_{274})$. A method for distinguishing betwe expressed and rectified oils consists in measuri the conjugated triene content before and af decolorisation with bleaching earth. For an e pressed oil there is a considerable increase (< times) on treatment, but for a rectified oil there only a small increase or a decrease.
L. A. O'Neill

2165. **Behaviour of Calabrian olive oils in Fitelson reaction for the detection of tea-seed oil.** A. Rome R. Cuzzocrea and E. Foti (Lab. Chim. Pro Reggio, Calabria, Italy). *Boll. Lab. Chim. Pro* 1960, **11** (3), 357–361.—The Fitelson test for t presence of tea-seed oil in olive oil is of limit value when applied to Calabrian olive oils, sin the pure oils themselves give similar colours whi may mask small percentages of added tea-seed o
J. I. M. Jones

2166. **Thiocyanate-value determination of t fatty acid composition of partially hydrogenat fats. II.** P. Möller and M. Gabrielsson (Res. La Karlshamns Oil Works, Karlshamn, Sweden *Fette, Seif., Anstrichmitt.*, 1960, **62** (10), 936–937. A paper-chromatographic method (Kaufmann an Arens, *Anal. Abstr.*, 1959, **6**, 2354) is used for t direct determination of 8,12-linoleic acid, which formed during the hydrogenation of linoleic aci containing oils, by the separation of its thiocyana adduct from that of the 9,12-linoleic acid. Son methods for determining fatty acid compositio are given. G. R. Whalley

2167. **Semi-micro method for the determinati of fatty acid composition by reversed-phase chr matography with gradient elution.** R. R. Brenn and O. Mercuri (Cátedra de Quím. Biol., Inst. Fisiol., La Plata). *An. Asoc. Quím. Argentin* 1959, **47** (4), 318–325.—A modification of th method of Howard and Martin (*Biochem J.*, 195 **46**, 532) is described. The chromatographic colum (30 cm × 1·5 cm) is charged with Hyflo Super-C previously treated with dimethyldichlorosilane an liquid paraffin according to the technique of Si and Hahn (*Anal. Abstr.*, 1954, **1**, 970), and mai tained at 36° in a thermostat. A concentratio gradient in the eluent is produced by means of tw communicating vessels. The first is filled wit acetone (200 ml) containing neutral liquid paraffi (20 mg per 100 ml) and the second, supplying th column, is filled with a 45% (v/v) solution of aceto in water, saturated with neutral liquid paraffin a 36°. Both solutions contain bromothymol blu (2 mg per 100 ml) and the mixture in the secon vessel is stirred with a stream of N. The sampl (1 to 40 mg of mixed fatty acids) is applied to th column as an emulsion in 45% aq. acetone and th

ate is collected in 2-ml portions and titrated with 1N-ethanolic KOH. Good separation, reproibility of volume retention and recovery of fatty ds is obtained with acids containing from 12 to C atoms. The modified technique is speedier and es a more compact elution curve than the ginal method. E. C. Apling

2168. **New method for the qualitative and quantiive analysis of synthetic antioxidants.** A. Seher tsch. Inst. f. Fettforschung, Münster, Westf.). *hrung*, 1960, **4** (5–6), 466–478.—Various methods ently published are discussed. It was found that n-layer chromatography is suitable for the separan and identification of all the usual antioxidants. e separation of polyhydric compounds can be proved by modifying the layers. Buffered layers e good results when stained with Gibbs reagent. antitative results can be obtained by means of mparison chromatograms. Antioxidants can be termined to an accuracy of ±5% by graphic erpolation. This method can also be used for e determination of vitamin E. I. Dickinson

2169. **Quantitative paper-chromatographic deternation of thiamine in food.** L. Wildemann ax-Planck-Inst. f. Ernährungsphysiologie, Dortmd). *Nahrung*, 1960, **4** (5–6), 497–511.—The ethod previously described for the determination thiamine in urine (Kraut and Wildemann, *Int. Vitaminforsch.*, 1956, **27**, 122; Kaiser and ildemann, *Ibid.*, 1956, **27**, 131) is applied to ods. Procedures for the extraction of thiamine om various types of food are described. The romatogram is developed in two directions with isopropyl alcohol - acetic acid soln. (pH 3·5) 1) and (*ii*) isopropyl alcohol - NaOH soln. H 9) (4:1). After the first development the per is dried at 60° and the one-dimensional romatogram is sprayed with alkaline $K_3Fe(CN)_6$ n., and the rest of the sheet with 0·1N-NaOH. ter the second development, strips of paper ntaining the spots of thiochrome and its phosoric acid ester are cut out, and each spot is ncentrated by the ascending technique with oxan - NaOH soln. (pH 9) (3:7). The spots are en dried, cut out, together with a similar blank ece of paper, and photographed under u.v. light. ie photographic apparatus and procedure and e method of calculation are described.

R. E. E.

2170. **Coulometric titration of L-ascorbic acid.** umikazu Kawamura, Kozo Momoki and Shigetaka zuki (Fac. of Engng, Yokohama Univ., Minami-, Yokohama). *J. Chem. Soc. Japan, Ind. Chem. ct.*, 1959, **62** (5), 629–631.—The coulometric ration of ascorbic acid (**I**) is satisfactorily carried t at pH 3 to 5 by means of iodine or bromine, oduced by the electrolysis at constant current to 10 mA) of 0·1M-KI or KBr. Metaphosphoric id (**II**) (0·25N) provides the best electrolyte. ssolved O must be removed by a current of N. ie method is applicable to the determination of VI (30 μg to 2 mg per 50 ml) by means of a known cess of **I**. Tervalent Fe (<50 times the molar nount of the Cr) is masked by **II**. K. Saito

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See also Abstracts—**1990**, Vitamin A in cod-liver . **2005**, Glycerol in wine. **2063**, ^{137}Cs, ^{89}Sr and Sr in milk, etc. **2206**, *o*-Hydroxydiphenyl in trus fruit. **2217**, Apparatus for N determination.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

2171. **Micro-determination of ozone in smog mixtures: nitrogen dioxide equivalent method.** B. E. Saltzman and N. Gilbert (U.S. Public Health Service, Cincinnati, Ohio). *Amer. Ind. Hyg. Ass. J.*, 1959, **20**, 379–386.—A new method is presented for conveniently and specifically determining low concn. of O_3 in polluted air, even in the presence of large amounts of other commonly occurring oxidising or reducing gases. Ozone was stoicheiometrically converted into, and determined as, NO_2 by the addition of controlled amounts of gaseous NO in N to the sample air stream and allowing a short reaction time. Better than 95% conversion was obtained when an excess of 1 p.p.m. of NO and 40 sec. reaction time were used. In the short time allowed, oxidation of NO by air and organic oxidants was negligible. Results for pure O_3 were in good agreement with those of an iodide reagent. For synthetic smog oxidants mixtures generated by the O_3 reaction with hex-1-ene, the method appeared specific for O_3, whereas the iodide reagent also responded to organic oxidants. Thus, the mixture could be differentiated into two oxidant components by simultaneous application of the two methods. Reducing gases such as SO_2 and H_2S did not appreciably interfere, even in a 100 to 1 ratio with O_3. The method should be readily adaptable to automatic recording of O_3 in smog without interference from associated pollutants. Chem. Abstr.

2172. **Spectrographic determination of beryllium in atmospheric dusts.** J. C. Cotterill (U.K.A.E.A., Research Group, C.37 Royal Arsenal, Woolwich, S.E.18). Analytical Method AERE-AM 56, 1960. 14 pp.—Air is drawn through a Fourstones filter-paper, which retains particles >0·5 μ in diam. After impregnation of the paper with a soln. containing Fe (7 mg) and Ni (1 mg) as nitrates it is dried, ashed and mixed with $(NH_4)_2SO_4$ (40 mg); pellets of this mixture are excited in a d.c. arc (7 amp.) between copper electrodes; a large automatic quartz spectrograph is used. The content of Be is derived from a comparison of the intensities of the lines Ni 2346·6 A and 2347·5 A and the line Be 2348·6 A. Full experimental details of the procedure, including the preparation of standards, are given. G. J. Hunter

2173. **Photo-electric determination of the turbidity of water.** E. L. Molt and L. H. Tio (Lab. Drinkwaterleiding, Rotterdam, Netherlands). *Chem. Weekbl.*, 1960, **56** (41), 553–557.—Several disadvantages were found in the use of silica, mastic, colloidal sulphur and kaolin as standards in the measurement of the turbidity of water. It is therefore proposed to replace methods in which a standard is used by a simple extinction measurement at 720 mμ in a 10-cm cell and to express the turbidity as the extinction per metre of cell length. The wavelength of 720 mμ is chosen in order to avoid corrections for the inherent colour of the sample, since at this wavelength the influence of colour on the turbidity measurement is negligible.

M. J. Maurice

2174. **Electrode for simplified field determination of chloride in ground water.** W. Back (U.S. Geol. Survey, Washington, D.C.). *J. Amer. Wat. Wks Ass.*, 1960, **52** (7), 923–926.—In this simple method,

a silver - silver chloride electrode, containing a billet of silver chloride with much of its surface exposed to the sample, is used in conjunction with a saturated calomel reference electrode and a standard pH meter. Measurements are made at the rate of 20 samples per hr., after standardisation. The theory, errors and limitations of the method are discussed.
O. M. Whitton

2175. **Suggested method for iodide determination.** J. R. Rossum and P. A. Villarruz (California Water Service Co., San José). *J. Amer. Wat. Wks Ass.*, 1960, **52** (7), 919–922.—A modification of the method of Lein and Schwartz (*Anal. Chem.*, 1952, **23**, 1507) is described. It takes less than an hour, and if several samples are tested in batches the average time per sample is only a few minutes. Iodide is used as a catalyst for the reduction of yellow acid ceric sulphate soln. to colourless cerous soln. by $NaAsO_2$ - NaCl soln.; the rate of fading is a function of the iodide concn. and is measured photometrically. The precision is $\simeq 3\%$. Interference is caused by reducing agents that react rapidly with ceric sulphate and by oxidising agents. Reducing agents are destroyed with $KMnO_4$. No interference is given at the concn. levels stated by Cl^- (5000 mg per litre), Na (3000) alkalinity (as $CaCO_3$) (1000), NO_3^- (750), Ca (250), Cd (10), Br^- (5), CN^- (5), F^- (5), Ag (1), or Fe (1). Mercury (>0·1 mg per litre) and Os interfere. O. M. Whitton

2176. **Titrimetric determination of nitrates in water.** D. Ceausescu (Timişoara Inst. of Hygiene, Romania). *Zhur. Anal. Khim.*, 1960, **15** (5), 646–648.—*Procedure*—To a sample containing 0·1 to 1 mg of NO_3^- add sufficient Ag_2SO_4 soln. (4·937 g per litre) to precipitate Cl^-, and evaporate to low bulk on a water bath. Cool and add 1 ml of phenoldisulphonic acid (prepared by heating 25 g of phenol with 150 ml of H_2SO_4 for 6 hr.). After 10 min. add H_2O (5 to 10 ml), almost neutralise the soln. with 6N-NaOH, then add 0·6N-NaOH until the soln. is yellow. Transfer to a stoppered flask, add 0·6N-NaOH (0·5 ml), dilute to 50 to 100 ml and add an equal vol. of benzene. Titrate with methylene blue soln. (0·001M), with vigorous shaking after each addition, until the aq. layer changes through green to blue. Standardise the methylene blue with 0·01M-KNO_3. J. W. Price

2177. **Determination of caesium-137, and strontium-89 and -90 in rain, lake and sea water.** U.K.A.E.A., Production Group (Windscale, Cumberland). U.K.A.E.A. Report PG 155 (W), 1960. 19 pp.—Rain- and lake-water samples are filtered; the residue is ignited, $HClO_4$ and HNO_3 are added and evaporated to dryness, and the product is leached with M-HCl. After the addition of carriers for Cs, Sr, K and Ca, conventional radiochemical separation procedures are used. Sea water is treated similarly except for a preliminary pptn. of the Ca and Sr as oxalates. β-Counting is used for all the nuclides and the assessment of ^{90}Sr is dependent upon the measurement of ^{90}Y. The procedure, for which full working instructions are given, can be applied conveniently to large samples (up to 50 litres) as no evaporation is involved; it has been used to analyse samples containing 2 to 1500 $\mu\mu$C of ^{137}Cs, 20 to 5000 $\mu\mu$C of ^{89}Sr, and 1 to 1000 $\mu\mu$C of ^{90}Sr. G. J. Hunter

2178. **Polarographic determination of low concentrations of nitrocyclohexane in waste water.** G. M. Vaĭnshteĭn and P. S. Papsueva (State Sci. Res. and Development Inst. of the Nitrate Industr and Products of Org. Synth.). *Zavod. Lab.*, 196(**26** (10), 1095–1097.—Nitrocyclohexane in a solr 0·25N in Na_2SO_4 and 0·25N in Na_2SO_3 gives a polarc graphic wave with $E_{\frac{1}{2}} = -0{\cdot}97$ V *vs.* the N.C.E With concn. >8 mg per litre, the wave height proportional to the concn. The minimum detect able wave corresponds to a concn. of 0·1 mg per litre G. S. Smith

2179. **Use of ion-exchange enrichment in th determination of trace elements in sea wate** R. R. Brooks (Dept. of Chem., Univ. of Cape Town) *Analyst*, 1960, **85**, 745–748.—The procedure depend on the failure of any of the major constituents o sea water to form anionic chloro-complexes in th presence of small amounts of HCl, whereas many of the trace elements form strong complexes. I the apparatus described, the sea water (0·25 ton was made 0·1N with HCl, sufficient bromine wate was added to adjust the concn. of this element t 10 p.p.m., and the water was allowed to flow slowl during 100 days through a small column of strongl basic anion-exchange resin (Amberlite R-400) The column was washed with 2N-HCl and then elute with 0·25N-HNO_3 (1 litre). The eluate was evapor ated and analysed spectrographically in a NaC matrix. The resin itself was ignited and the ash analysed in the same way. A blank was also carrie out. Distinct lines were detected for Au (2676 A in the resin fraction and for Bi (3067 A), Cd (3261 A and Zn (3345 A) in the eluate fraction. Thallium appeared to be absent and the Zn line was too diffuse for accurate measurement. The Sb line (3267 A) was used as an internal standard, and working graphs were prepared for the determinatio of Cd, Au and Bi. The abundances found (mg pe ton) were Au, 0·009; Bi, 0·017; Cd >0·02.
A. O. Jones

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

2180. **Application of direct photometry to agricul tural analysis.** R. O. Scott (Macaulay Inst. fo Soil Res., Craigiebuckler, Aberdeen, Scotland) *J. Sci. Food Agric.*, 1960, **2** (10), 584–592.—The applications of two direct-reading spectrometer (porous-cup spark method) to the analysis o agricultural materials are described. With the use o (*i*) a two-channel instrument for the determinatio of 0·3 to 24 p.p.m. of Mg in acetic acid or ammon ium acetate extracts of soil and HCl extracts o plant material, the coeff. of variation for 40 deter minations on a soln. containing 1·8 p.p.m. of M was about $\pm 2{\cdot}0\%$; the major composition o agricultural samples does not affect the determina tions. With the use of (*ii*) the eleven-channe Hilger Medium Direct Reader for the determinatio in soil extracts of 0·1 to 24 p.p.m. of Cu, 1 to 40 p.p.m. of Mn and 1 to 50 p.p.m. of Zn, the coeff of variation are $\pm 1{\cdot}10\%$, $\pm 1{\cdot}64\%$ and $\pm 7{\cdot}01\%$ respectively. A tentative method, in which pelleted rotating disc is used, is given for the deter mination of Zn, B, Mn and Cu in plant materials with coeff. of variation of $\pm 1{\cdot}9\%$, $\pm 2{\cdot}8\%$, $\pm 3{\cdot}3\%$ and $\pm 5{\cdot}4\%$, respectively. I. Dickinson

2181. **Analysis of the structural carbohydrate of herbage.** I. H. Bath (Nat. Inst. for Researc in Dairying, Shinfield, Reading, England). *J. Sci Food Agric.*, 1960, **2** (10), 560–565.—A method i

posed for the fractionation of the carbohydrates plant material by successive extractions with nol - benzene, 95% ethanol and hot water, owed by delignification and extraction of the ated holocellulose with KOH soln. to yield icellulose and cellulose. The polysaccharides ne hot water extract, hemicellulose and cellulose hydrolysed, and the constituent monosac-rides are separated by paper chromatography determined spectrophotometrically.

I. Dickinson

182. **Losses of material during moisture determinations on cereals.** H. Bolling (Bundesforungsanst. f. Getreideverarbeitung, Detmold, many). *Getreide u. Mehl*, 1960, **10** (9), 102–—Methods of determining moisture in cereals h sound and spoiled) are critically reviewed. ght losses, obtained by heating at various p. and pressures, are compared with moisture erminations by the Karl Fischer method and n a direct moisture-absorption method. The ure of non-aqueous substances lost during ting is investigated by paper-chromatographic hods, on paper impregnated with high-boiling rocarbons, with acetic acid - water as the mobile se and $K_4Fe(CN)_6$ as spray reagent. The stances are mainly oleic and linoleic acids. references.)

J. V. Russo

183. **Examination of agricultural products by formic acid method for determining cellulose, ch and protein.** E. Lehmann and H. Birsgal. *PflErnähr. Düng.*, 1960, **88**, 1–13.—Plant terial is de-fatted with ethyl ether and 1 g of ground product is treated with 25 ml of formic l (99 to 100%) for 18 to 20 hr. in a centrifuge e. After centrifuging for 15 min. and washing ce with formic acid, the residue is stirred with re formic acid and set aside overnight. The nylcellulose is washed thrice with ether, dried weighed. From 25 ml of the combined formic l extracts, formylstarch is pptd. with 125 ml of cial acetic acid plus 2 ml of benzene. After min. the suspension is centrifuged and the due is washed twice with portions of the de-ted supernatant liquid and twice with acetic l. The formylstarch is washed with ether, ed and weighed. Formylprotein in the com-ed supernatant liquids from the formylstarch aration is pptd. by further addition of 50 ml ether, benzene and light petroleum, separated centrifuging, washed twice with the clear ernatant liquid and thrice with ether, dried and ghed. Amino-acids, peptides and low-mol.-wt. bohydrate materials remaining in soln. after ioval of protein may be recovered by vacuum tillation. Hydrolysis of the formylcellulose is cted by heating for 3 min. with 1·6% NaOH n., acidifying with acetic acid and adding thanol. The ppt. is washed with methanol, ed and weighed. The starch compound is pended in 10 ml of water plus 4 ml of 5% NaOH n. for 6 hr. The liquid is then acidified with % acetic acid, and 15 ml of methanol is added. e ppt. is collected by centrifuging, washed twice h methanol and twice with ether (50 ml) and ed at 110°. The formylprotein is stirred into o 5 ml of 2% piperidine soln. plus 10 ml of water. er 3 hr., 1 to 5 ml of 10% acetic acid is added l the protein is pptd. with 60 ml of acetone for min., then centrifuged, washed with acetone, n with ether, dried at 100° and weighed.

A. G. Pollard

2184. **Spectrophotometric determination of dicyandiamide in soil.** A. Pawlik. *Z. PflErnähr. Düng.*, 1960, **89**, 181–187.—The u.v. absorption spectrum of dicyandiamide differs from those of its decomposition products (guanidine, guanylurea, urea, nitrate) in soil sufficiently clearly to serve for its determination in aq. extracts of soil. The max. absorption is at 215 mμ.

A. G. Pollard

2185. **Absorptiometric determination of fluoride in grass.** U.K.A.E.A., Production Group (Chem. and Met. Services, Springfields, Lancs.). U.K.A.E.A. Report PG 144 (S), 1960. 8 pp.—Full details of the procedure are given. The dried grass (25 g) is ashed at 600° with Mg acetate. After the addition of U_3O_8 (1 g) to the residue, steam is passed over the mixture at 1000°. The pyrohydrolysate (240 ml) is collected in a vessel containing NaOH soln. The content of F is determined, after adjustment of the pH of the soln. to 4·5, by adding a standard amount of aluminium - Solochrome cyanine (C.I. Mordant Blue 3) complex and measuring the reduction in extinction. The coeff. of variation is ≃8% in the range 5 to 350 p.p.m. of F.

G. J. Hunter

2186. **Contamination problems in soil and plant analysis.** R. L. Mitchell (Macaulay Inst., Craigiebuckler, Aberdeen, Scotland). *J. Sci. Food Agric.*, 1960, **2** (10), 553–560.—The contamination of samples during transport in unsuitable containers or during drying and sieving is investigated. Means of assessing the severity of contamination of plants by soil and its possible effect on analytical results by determination of the apparent titanium content of the plant material are described. Contamination arising in the laboratory can be minimised by stringent precautions during working and the use of purified reagents.

I. Dickinson

2187. **Micro-determination of cation-exchange capacity and total exchangeable bases [in soil].** L. C. Blakemore and A. J. Metson. *Soil Sci.*, 1960, **89**, 202–208.—The standard ammonium acetate procedure is adapted for use with 20-mg samples of soil; a miniature leaching-tube (Pyrex glass, internal diam. 5·5 mm) is used, the lower separable section (plastic sleeve) being drawn out to an orifice (1·5 mm in diam.). Total bases in the leachate are determined by evaporation to dryness, ignition at 500°, dissolution of the resulting carbonates in standard acid and back-titration. The leached soil is washed with 90% (w/w) ethanol containing 0·5 ml of N-aq. NH_3 per litre and finally with 95% ethanol free from NH_3. The NH_3 in the residual soil is determined by the Conway diffusion technique.

A. G. Pollard

2188. **Determination of exchangeable cations in soils with the Beckman Model B flame spectrophotometer.** P. F. Pratt and G. R. Bradford. *Soil Sci.*, 1960, **89**, 342–346.—Aliquots of the neutral ammonium acetate leachates from soil are passed through a column containing about 25 ml of Cl^--saturated Dowex 1-X8 resin (50 to 100 mesh) to remove PO_4^{3-} and SO_4^{2-}. The first 20 ml of percolate is discarded and the following 15 to 20 ml serves for determining Ca, Na and K (or 50 to 70 ml if Mg is also to be determined). Calcium is determined flame-photometrically (acetylene - O flame) at 423 mμ, Na (H - O) at 589 mμ, and K at 768 mμ. Magnesium is separated from the percolate by pptn. with NaOH in the presence of mannitol (methyl red end-point). The ppt. is separated centrifugally, washed with aq. NaOH - mannitol,

dissolved in HCl and determined by spectrophotometry at 372 mμ. The aliquot of percolate from the resin column should not contain in 25 ml more than 300 p.p.m. each of Ca, Na and K, or 200 p.p.m. of Mg. A. G. POLLARD

2189. **Determination of exchangeable hydrogen in soils by a titration method.** T. L. Yuan. *Soil Sci.*, 1959, **88**, 164–167.—The air-dry soil sample (20 g) is stirred with 50 ml of N-KCl and after 30 min. the mixture is filtered. The residual soil is leached three more times with 50 ml of N-KCl, draining thoroughly each time. To the combined filtrates are added 10 ml of 4% NaF soln. and, after stirring, 10 drops of 0·1% phenolphthalein soln., the mixture being then titrated with 0·1N-NaOH. The vol. of alkali used is equivalent to exchangeable H^+. Alternatively, the combined filtrates are titrated directly with 0·1N-NaOH to phenolphthalein, stepwise until the pink colour persists (more indicator may be needed). The vol. of alkali used is equivalent to exchange acidity. After titration, the pink colour is discharged by one drop of 0·1N-HCl, 10 ml of 4% NaF soln. is added and the liquid is titrated with 0·1N-HCl till no pink colour returns. The vol. of acid used is calculated as exchangeable Al; the exchangeable H^+ is given by the difference between exchange acidity and exchangeable Al. A. G. POLLARD

2190. **Determination of soil water by dielectric measurement of dioxan extract.** H. W. van der Marel. *Soil Sci.*, 1959, **87**, 105–119.—The soil is shaken for 10 min. with dioxan. The suspension is filtered and the dielectric constant of the filtrate is determined. Standard mixtures of dioxan and water serve for calibration. Soil salinity does not affect the measurement. A. G. POLLARD

2191. **Determination of the cement or lime content of cement- or lime-stabilised soil.** Road Research Laboratory (Road Res. Lab., Harmondsworth, Middx., England). *Road Note*, 1960, No. 28. 12 pp. —Rapid procedures, dispensing with filtrations, for the usual (*i*) EDTA and (*ii*) flame-photometric methods are given. In method (*i*), the content of cement or CaO in the sample is calculated from the contents of Ca plus Mg in the soil, stabilised soil and cement (or CaO). These are determined, after extraction of the sample with 6N-HCl and pptn. of the mixed hydroxides, by titration at pH 10 with EDTA (disodium salt), Eriochrome black T being used as indicator. A correction is made for any Ca co-pptd. with the mixed hydroxides; the error arising from variable amounts of Mg can be minimised by adopting the titration procedure of Patton and Reeder (*cf. Anal. Abstr.*, 1956, **3**, 2982). In method (*ii*), the sample soln. (prepared from 5 g of the soil-cement or -lime and free from Fe and Al) is sprayed through the flame of an EEL photometer which has been calibrated with reference samples of stabilised soil. Provided that the bulk samples have been well mixed and finely ground, the error should not exceed 0·2%.
W. J. BAKER

2192. **Photometric determination of total phosphorus in soil.** M. L. Tsap and G. L. Linnikova. *Pochvovedenie*, 1960, (2), 102–107; *Ref. Zhur., Khim.*, 1960, (18), Abstr. No. 73,112.—Two variants are described of the photometric determination of P in soil as the blue molybdophosphate complex, in which the reducing agent is a mixture of Na_2SO_3 with a ferrous salt, or hydrazine sulphate.
C. D. KOPKIN

2193. **Ammonium lactate method for determin readily available phosphate in soil contain carbonates.** H. Riehm (Staatl. Landw. Versuchs Forschungsanst., Augustenberg/Baden-Württe berg). *Agrochimica*, 1958, **3**, 49–65.—The sam (5 g) of air-dried sieved (2 mm) soil is shaken 4 hr. at 20° ± 1° with 100 ml of a soln. contain ammonium lactate (0·1N) and acetic acid (0·4N) pH 3·7. Phosphate in the filtrate is determined the molybdenum blue method. On this ba soils yielding 20 mg of P_2O_5 per 100 g are graded "rich", those with 11 to 20 mg as "adequate" those with 0 to 10 mg of P_2O_5 as "deficient."
A. G. POLLARI

2194. **Manometric determination of calcite dolomite in soils and limestones.** S. I. M. Skin R. L. Halstead and J. E. Brydon (Canad. De Agric., Ottawa). *Canad. J. Soil Sci.*, 1959, 197–204.—The sample, sufficient to give a fi manometer reading of 3 to 4 in. of mercury, placed in a waxed paper cup and floated on 30 of 4N-HCl in a 700-ml wide-necked reaction bo connected to a mercury manometer. The react is carried out with the bottle clamped in a sha and immersed in a water bath at 25°. The log the amount of unreacted CO_2 is plotted agai time. The rapid decomposition of calcite gi place to the slower reaction of dolomite and graph approaches a straight line, extrapolation which to zero shows the amount of dolom originally present. The amount of calcite calculated by difference from the total CO_2 obtain
A. G. POLLARI

2195. **Assessing the zinc status of soils us acid-extractable zinc and "titratable alkalini values.** J. L. Nelson, L. C. Boawn and F. Viets, jun. *Soil Sci.*, 1959, **88**, 275–283.—Ac extractable Zn is determined in 2 g of air-dr soil placed in a glass-stoppered centrifuge tube w 20 ml of 0·1N-HCl. After being shaken for 5 m the mixture is centrifuged and the residual soi extracted twice more with the acid. Zinc is de mined in an aliquot of the total extract by A.O.A.C. dithizone method. "Titratable alkalini is defined as the amount of acid (ml per 10 needed to produce a pH of 5 in the soil. graphical relation between acid-extractable Zn a "titratable alkalinity" permits differentiation tween soils deficient in, and those adequat supplied with, Zn. A. G. POLLARI

2196. **Determination of free iron [oxides] in pa soils.** T. Asami and K. Kumada. *Soil and Pl Food, Tokyo*, 1959, **5**, 141–146.—To the sample to 2·0 g) of air-dried soil are added 3 g of Na_2S and 100 ml of 0·02M-EDTA. The mixture maintained at 70° for 15 min., with occasio shaking, and filtered, the residue being washed th times with 1% aq. NaCl. An aliquot of the co bined filtrates is transferred to a 25-ml flask toget with 1 ml of 5% aq. hydroxyammonium chlor soln., 2 ml of 1% 1,10-phenanthroline and 1·5 of buffer soln. (Na acetate, 172 g in 500 ml, p glacial acetic acid, 6 ml). After dilution of the sc to vol. and setting aside for 30 min. the extinctio measured at 508 mμ. A. G. POLLARI

2197. **Determination of nitrogen in soil by Kjeldahl method.** J. M. Bremner. *J. Agric. S* 1960, **55**, 11–33.—Among variants of the origi method examined, the A.O.A.C. method ga results 10 to 37% lower than other metho

h, in general, gave similar values. As catalysts, nd Hg were superior to Cu. Loss of N when g Se as catalyst was associated with proportions e $>0{\cdot}04$ g per 3 ml of H_2SO_4 or with >1 g of $)_4$ together with 0·02 g of Se per 3 ml of acid. eneral, loss of N during digestion occurred at). $>400°$, this being mainly controlled by the unt of K_2SO_4 or Na_2SO_4 used, 1·66 g of K_2SO_4 nl of acid giving rise to temp. $>400°$. Micro- macro-techniques gave similar results provided in the former, the sample was ground to a 100-mesh sieve. A. G. Pollard

98. **Colorimetric determination of soil organic er.** E. R. Perrier and M. Kellogg. *Soil Sci.*, , **90**, 104–106.—The sample (exactly 0·5 g) of ried soil (100 mesh) is placed in a 75-ml test- to which are added 10 ml of N-$K_2Cr_2O_7$ and l of H_2SO_4 slowly from a burette. After mixing solutions the tube is placed in boiling water 5 min. and then cooled. The solution is diluted litre and an aliquot (1 ml) is transferred to a ml flask; 6N-H_2SO_4 (3·3 ml) and 1 ml of sym.- enylcarbazide reagent are added and the whole luted to the mark. The extinction of the violet ur produced in the test solution is measured 40 mμ within 10 min. of preparation.
A. G. Pollard

99. **Measurement of carbohydrates in soil olysates with anthrone.** R. H. Brink, jun., Dubac and D. L. Lynch. *Soil Sci.*, 1960, **89**, -166.—The technique of Morris (*Science*, 1948, 254) is adapted. The sample (normally 5 g) of lried soil is hydrolysed on a steam bath for 24 hr. 3N-H_2SO_4 (1 ml per g of soil). The hydrolysate, e still hot, is filtered through a medium-grade ered glass filter and the solid is washed with water (10 ml per g of soil). The cooled com- d filtrates are diluted to a suitable vol. An uot (5 ml) is placed in a test-tube (150 mm × nm) (size is important) to which is added, with ring, 10 ml of anthrone reagent [0·2% in 95%) H_2SO_4], freshly prepared. The resulting green ur is measured spectrophotometrically at mμ after 15 min. A blank (5 ml of water and nl of reagent) is necessary. Standards made n glucose must be prepared daily.
A. G. Pollard

200. **Determination of free phosphoric acid in erphosphate.** F. Yamazoe. *Soil and Plant d, Tokyo*, 1960, **5**, 161–166.—A 2-g sample of ed (0·5 mm) material is shaken for 30 min. with ml of acetone - ether (1 : 1), then filtered through paper. An aliquot (50 ml) is evaporated to dryness the residue is dissolved in about 50 ml of hot er and titrated with 0·1N-NaOH (standardised n sulphamic acid to bromothymol blue) to the end-point (NaH_2PO_4) with dimethyl yellow , further, after addition of phenolphthalein, to nal and clear end-point (Na_2HPO_4).
A. G. Pollard

201. **Rapid, accurate method for the determina- of free phosphoric acid in superphosphate.** Balica and P. Oprică. *Rev. Chim., Bucharest*, 0, **11** (9), 527–528.—The method is based on the uction of oxidising agents by iodides in the sence of H^+, according to the reaction—

$$BrO_3^- + 6I^- + 6H^+ \rightleftarrows Br^- + 3I_2 + 3H_2O.$$

cedure — The finely ground superphosphate ($\simeq 2{\cdot}5$ g) is triturated with 25 ml of H_2O. When the solid has settled, the supernatant liquid is decanted through a fine filter. This operation is repeated thrice. The undissolved residue is trans- ferred to a filter and washed until the vol. of total filtrate is 200 ml. A 50-ml aliquot is diluted to 150 ml, treated with 50 ml of 0·1N-$KBrO_3$ and 2·5 to 3 g of KI. The mixture is shaken for 5 min. and titrated with 0·1N-$Na_2S_2O_3$, with starch as indicator. The errors compared with the classical NaOH method are $<0{\cdot}05\%$. H. Sher

2202. **Determination of dry matter and volatiles in silage.** P. McDonald and W. A. Dewar (Edinburgh School of Agric., West Mains Road, Edinburgh, Scotland). *J. Sci. Food Agric.*, 1960, **2** (10), 566–570.—An apparatus designed to collect the volatile constituents produced during the drying of food was used to investigate those of silages dried at 100°. In 28 silages examined, the mean volatility of acetic acid was 87·9% and of butyric acid 89·4%. Lactic acid was volatile from all samples, the volatility ranging from 1·4 to 16·4%. Appreciable losses of N occurred during drying of silages with a high pH. I. Dickinson

2203. **Determination of rotenone in *Lonchocarpus*.** D. V. Richmond. *Ann. Rep. Agric. Hort. Res. Sta. Bristol*, 1958, [1959], 99–101.—Standard methods based on the direct crystallisation of the rotenone - CCl_4 solvate give results $\simeq 10\%$ lower than the true values, possibly because other components of the benzene extract suppress the crystallisation of the solvate. The root (20 g) is extracted by hot per- colation with benzene for 3 hr., and the vol. of the cooled extract is adjusted to 50 ml. Ethyl ether (50 ml) is added and the mixture is extracted successively with 50-ml portions of 2, 5 and 5% KOH soln. in a separating-funnel. After removal of each alkaline layer, water is added immediately to lower the residual alkalinity of the liquid remain- ing in the funnel. The alkaline extract is washed with benzene - ether which is returned to the main soln., which is then acidified to litmus with dil. HCl, washed with water and dried over Na_2SO_4, and the solvent is distilled off under reduced pressure. The resin is dissolved in five times its wt. in ml of CCl_4 (previously saturated with rotenone at 0°) and the soln. is maintained at 0° overnight. The solvate is filtered off on pre-cooled apparatus, washed with a small amount of CCl_4 at 0°, dried at 40° and weighed. A. G. Pollard

2204. **Determination of residues of gamma-BHC.** J. A. Pickard. *Ann. Rep. Agric. Hort. Res. Sta. Bristol*, 1958, [1959], 93–95.—The method of Schecter and Hornstein (*Anal. Chem.*, 1952, **24**, 544) is modified. Plant material is extracted with dichloromethane (3 hr.) and the cold extract is passed through a column of activated Al_2O_3 (Brockman activity 1) and washed through with several portions of dichloromethane. The solvent is removed by a current of dry air at 30° to 35° and the final 1 ml by evaporation at room temp. The residue is dissolved in 10 ml of warm acetic acid and transferred via a cotton-wool filter, washing with 4, 4 and 3 ml of acetic acid, to the dechlorination apparatus. After addition of zinc (4 g), the mixture is boiled for 2·5 hr. in a stream of CO_2 to transfer the benzene formed to the nitration chamber containing 4 ml of HNO_3 - H_2SO_4 (1 : 5). The resulting nitration mixture is diluted to 25 ml, transferred to a flask with washings (30 ml),

neutralised (litmus) with 25% KOH soln., diluted to about 100 ml, and shaken (1 min.) with 20 ml of isobutyl methyl ketone. The two layers are separated and ethyl methyl ketone (9 ml) is added to an aliquot (⩾10 ml) of the organic phase followed by 1 ml of tetramethylammonium hydroxide reagent (8 ml of 25% soln. diluted to 100 ml with methanol). The extinction is measured within 5 min. at 565 mμ in a 2-cm cell. Extreme purity of reagents is essential. A. G. POLLARD

2205. **Determination of microgram quantities of dieldrin.** E. J. Skerrett and E. A. Baker. *Ann. Rep. Agric. Hort. Res. Sta. Bristol*, 1958, [1959], 95–99.—The test soln. (up to 100 μg of dieldrin in 5 ml of specially purified benzene) is heated for 30 min. at 78° with 1 ml of a mixture (9:1) of benzene and BF_3 - ether complex and cooled in ice-water. Saturated $NaHCO_3$ soln. is added slowly with cooling and the mixture is transferred to a separating-funnel with 5 ml of benzene. When no more CO_2 is liberated, the aq. layer is discarded, and the benzene soln. is washed with water, dried over Na_2SO_4 and evaporated to dryness. The residue is dissolved in 0·5 ml of ethanol, and 0·1 ml of freshly prepared 2:4-dinitrophenylhydrazine soln. [10 mg in 5 ml of 20% (v/v) H_2SO_4] is added. After 15 min. the mixture is transferred to a separating-funnel with 5 ml of benzene, and washed successively with HCl (10 ml), 10N-NaOH (5 ml), HCl, and water (20 ml). The dried (Na_2SO_4) benzene soln. is diluted to 7·5 ml and mixed with ethanol (2·5 ml) and 1 drop of 25% tetraethylammonium hydroxide soln., and after 1 min. the colour is matched against standards at 440 mμ. A. G. POLLARD

2206. **Determination of o-hydroxydiphenyl. I. Paper-chromatographic detection of o-hydroxydiphenyl.** H. Thaler and H. Günder (Dtsch. ForschAnst. f. Lebensmittelchem., München, Germany). *Dtsch. LebensmittRdsch.*, 1960, **56** (9), 262–264.—The detection and semi-quant. determination of *o*-hydroxydiphenyl residues in the peel of citrus fruits in amounts near the tolerance level of 10 p.p.m. are described. *Procedure*—Peel the sample of oranges, lemons or grapefruit (500 g). Coarsely mince the peel and shake half the total in a stoppered flask with 2N-H_2SO_4 (15 ml) and cyclohexane (150 ml) for 1 hr. Decant the cyclohexane and extract an aliquot (50 ml) with 0·1N-NaOH (2 × 10 ml), shaking for 3 min. each time. Acidify the combined extracts with N-HCl (3 ml) and shake with cyclohexane (3 ml) for 3 min. Separate the organic phase and apply from 0·05 to 0·1 ml to a strip of S. & S. 2043b paper and develop for 6 hr. at 20° to 22° with a mixture of methanol, formic acid and water (5:1:5). Spray the dry chromatogram with a neutral diazotised soln. of Na 1-naphthylamine-7-sulphonate, dry and re-spray with N-HCl. The *o*-hydroxydiphenyl spot is distinguished from spots of compounds natural to the fruit by a citron-yellow colour in daylight which appears dark under u.v. light. Semi-quant. determinations may be made by comparison with standards treated similarly. E. C. APLING

See also Abstracts—**1942**, Uranium in grass. **2061**, Sulphate in dried pasture. **2063**, ^{137}Cs, ^{89}Sr and ^{90}Sr in plants. **2151**, Org. F residues in blackcurrants. **2152**, DDT and hexachlorocyclohexane in flour and grain.

5.—GENERAL TECHNIQUE AND APPARATUS

General

2207. **Ceramic mills in a paint mixer for prep tion of multiple rock samples.** A. T. Myers W. H. Wood (U.S. Geol. Survey, Denver, Co *Appl. Spectroscopy*, 1960, **14** (5), 136–13 Geological samples are ground without contam tion by using vials and ball pestles made f ceramic containing 96% of alumina. Each has a capacity of 50 ml, and takes 17 to 18 m sample and 3 balls of § in. in diam. Six such v are mounted in a mixer giving 725 cycles per m of an eccentric shaking motion. Normal grin time is about 20 min. and particle size, with gra for example, is 98·5% of −270 mesh, and 95 of −325 mesh. P. T. BEAL

2208. **Improvements relating to the analysis liquids.** Technicon International, Ltd. Brit. 855,555; date appl. 3.2.58. U.S.A., date a 6.3.57.—When performing continuous analyses liquids in a system wherein the liquid to be analy is mixed with a primary processing liquid, w the liquids flow through conducting tubes, and or other inert gas is introduced into the liq stream and divides it into alternate segments liquid and air, for preventing the liquid from adhe to the walls of the tubes, better cleaning actio obtained on the walls of the tubes if the prim processing liquid is not introduced before the and the liquid under analysis have been brou together. Moreover, when a record is made of analysis of certain liquids, *e.g.*, blood, spur peaks, which are otherwise liable to occur, eliminated. J. M. JACOB

2209. **Balance with automatically levelling be** A. V. Shlȳkov. U.S.S.R. Pat. 128,633 (15.5.1960 To one end of the beam is attached a non-magn horizontal tube, *e.g.*, of glass, containing a s ball, which is in the magnetic field of a horses magnet, the arms of which embrace the tu the magnet is fixed to a carriage which itself move under the action of a screw-thread rotated a reversible electric motor, which is actuated contacts on the beam. A pen attached to carriage records the changing position of the s ball during continuous weighing, and therefore weight, on a rotating drum. The apparatus illustrated. C. D. KOPKI

2210. **One-mark volumetric flasks.** Brit Standards Institution (2, Park Street, London, W B.S. 1792:1960. 10 pp.—Standards are given flasks having 5 to 2000 ml capacity.

2211. **Automatic titrator for chemical analy** M. A. Veksler. *Zavod. Lab.*, 1960, **26** (9), 1146–11 —A versatile automatic titrator is described. G. S. SMIT

2212. **Gas analysis.** M. T. Borok. U.S.S Pat. 128,202 (28.4.1960).—The presence and con of a component is found by the thermal effect w the component dissociates. The gas is heated thermal resistances in a bridge circuit; the magnit of the maximum heat transfer as the hot dissocia molecules diffuse to a cold body is directly p portional to the concn. of the dissociating co ponent. If the dissociation takes place in seve directions or in several stages, depending on

perature, the maxima are measured for each ess or step on the pure component, and these used in the determination of the component in mixture (analogously to individual spectra in trographic analysis). No specific examples are n. C. D. KOPKIN

213. **Gas analyser.** E. A. Abov, D. B. Zvorykin V. S. Kondrashov. U.S.S.R. Pat. 128,656 5.1960).—A component of a gas is determined comparison of the temp. of the gas before and r its catalytic oxidation. The temperatures are sured by thermistors, and the catalytic zone is rated by a vacuum or gas space from the inner l of the water jacket which acts as a thermostat id the catalytic zone. The apparatus is trated, but no specific examples are given. C. D. KOPKIN

214. **Apparatus for the analysis of gases [particularly the detection, or determination of the centration of oxygen in gases].** United Kingdom mic Energy Authority London [Inventor: J. Allsopp]. Brit. Pat. 856,628, date appl. 57.—The apparatus (*cf.* Brit. Pat. 707,323) prises a galvanic cell to which the gas is supplied, gen being absorbed at the cathode to produce electric current which is a function of the oxygen cn. in the gas and is used to operate indicating recording means, *e.g.*, a galvanometer. The node (silver) is of sheet material formed into a se roll providing a spiral gas path, the axis of ch is vertical. The upper edge of the cathode ecured in a gas-tight manner to the cover of the . The anode (a pellet of lead cast on the end of latinum wire) is contained in a tube extending axially within the cathode and having its bottom closed by a porous disc. J. M. JACOBS

215. **Apparatus for determining the presence of dising agents [in gases].** Mine Safety Appliances Brit. Pat. 854,357, date appl. 1.4.59. U.S.A. e appl. 3.4.58.—The presence of an oxidising nt, including Cl, Br and NO_2, in a gas, *e.g.*, air, letected by passing the air through a tube coning an inert granular solid (silica gel) carrying to 300 mg per 10 cu. cm of gel of a reagent sisting of *NNN'N'*-tetraphenylbenzidine or *NN'*-ethyl-*NN'*-diphenylbenzidine and, when the sence of NO_2 is to be detected, H_2SO_4, HCl, ClO_4, trichloroacetic or toluene-*p*-sulphonic acid. e presence of the oxidising agent is indicated by formation of a blue colour. The method can be apted to the quant. determination of the gases. J. M. JACOBS

2216. **Improved apparatus for the quantitative tillation of steam-volatile substances.** N. Antonpoulos (Inst. f. Fischverarbeitung, Bundesforungsanst. f. Fischerei, Hamburg, Germany). *LebensmittUntersuch.*, 1960, **113** (2), 113–116.—eam is generated in a wide-mouthed round flask ually 2-litre) and, during the main operation, sses through a tube leading to the bottom of a pe-shaped vessel containing the sample, which ssel fits into the mouth of the flask so that its ntents are heated by the boiling water, and is nnected with a still-head and condenser. During e preliminary heating, a tap-funnel situated near e top of the flask is kept open to prevent steam ndensing in the sample tube. This apparatus gives, comparison with the conventional arrangement, reased yields of volatile matter in smaller vol. of tillate. P. S. ARUP

2217. **Determination of nitrogen or crude protein by means of an "improved apparatus for the quantitative distillation of steam-volatile substances."** N. Antonacopoulos (Inst. f. Fischverarbeitung, Bundesforschungsanst. f. Fischerei, Hamburg, Germany). *Z. LebensmittUntersuch.*, 1960, **113** (2), 116–117.—The apparatus previously described (*Anal. Abstr.*, 1961, **8**, 2216) is modified for use in Kjeldahl distillations by the insertion of a tap-funnel into the top of the still-head for the purpose of introducing the NaOH soln. P. S. ARUP

2218. **The azeograph—a new apparatus for analytical azeotropic distillation.** J. Pokorný and B. Melichar (Dept. Pharm. Chem., Pharm. Fac., Brno, Czechoslovakia). *Českosl. Farm.*, 1960, **9** (8), 409–412.—A new modification of the apparatus of Melichar and Pokorný (*Chem. Listy*, 1956, **50**, 844) with automatic registration of distillation curves at 760 torr is described. The procedure can be carried out on the semi-micro scale. A thermistor is used for the detection of the temp. changes, and the curves are automatically recorded on photographic paper. The apparatus can be used for the identification and determination of volatile compounds and for the study of the relation between the azeotropic behaviour of compounds and their chemical constitution. J. ZÝKA

2219. **High-frequency furnace for organic elementary analysis.** Eisaku Kimura (Fac. of Pharm. Sci., Tokyo Univ., Hongo, Tokyo). *Japan Analyst*, 1959, **8** (12), 767–773.—The use of the Hartley circuit has been examined. The energies of the four main parts (the oxygen-purifying furnace, the furnace for combustion of the sample, the oxidation-accelerating furnace and the constant-temperature furnace) are regulated by changing the capacity of condensers attached to each part. The combustion furnace in particular is automatically controlled by changing the temp., according to the gas pressure evolved on decomposition of the sample. K. SAITO

2220. **Simple transistor-operated oven-temperature regulator.** E. R. Pike and J. F. Cochran (Res. Lab. of Electronics, M.I.T., Cambridge, Mass., U.S.A.). *Rev. Sci. Instrum.*, 1960, **31** (9), 1005–1007.—The circuit described regulates the temperature of an oven of max. power dissipation 100 W and max. temp. 1500° with a variation of $<1°$ over long periods. The performance of the regulator can be further improved, particularly against mains-voltage fluctuations, by the inclusion of a simple transistor amplifier (described). G. SKIRROW

See also Abstracts—**1906**, H.f. combustion furnace. **1917**, Apparatus for O in gas - air mixture. **1989**, Apparatus for H_2O in org. compounds. **2015**, Non-aqueous cerimetry.

Chromatography, ion exchange, electrophoresis

2221. **Quantitative evaluation of paper chromatograms of inorganic ions by X-ray fluorescence.** E. Jackwerth and H.-G. Kloppenburg (Inst. f. Spectrochem. u. angew. Spectroscopie, Dortmund-Aplerbeck). *Naturwissenschaften*, 1960, **47** (19), 444.—Spots on strip chromatograms are identified and quant. evaluated with the aid of an attachment

to the Siemens X-ray spectrometer; the strip is passed at a constant slow speed through the X-ray beam incident at 45°. For R_F determination, the characteristic angle of inclination for max. fluorescence is measured. For the quant. evaluation of small spots (> 10 μg), the total fluorescence is determined with a scintillation counter; for amounts > 100 μg, the fluorescence is recorded as the strip passes through the X-ray beam, and the area under the curve is measured. The coeff. of variation in the separation of $\simeq 5$ μg of Ga and Zn is $< 5\%$. The lower limit of determination is 1 μg of Ga and Zn. J. P. STERN

2222. **Use of chlorine in the detection of [amino- and imino-] compounds on paper chromatograms.** C. G. Greig and D. H. Leaback (Biochem. Dept., Inst. of Orthopaedics, Brockley Hill, Stanmore, Middlesex, England). *Nature*, 1960, **188**, 310–311.—To detect amino- and imino-compounds, the dried chromatograms are exposed to an atmosphere of Cl, followed by aeration to remove background Cl and detection of the resulting chloramines by liberation of iodine from a starch - KI reagent (*cf.* Rydon and Smith, *Brit. Abstr. C*, 1953, 557). The chlorination and aeration are difficult to standardise. To facilitate the method the following procedures have been used—(*a*) the chromatogram is sprayed with NaOCl soln. (10 to 14%) diluted 1:150, then left for 45 min. at room temp.; (*b*) the chromatogram is sprayed with NaOCl soln. diluted 1:25, left for 45 min. and then heated for 5 min. at 55°; (*c*) the chromatogram is sprayed with $KClO_3$ - HCl reagent ("The Merck Index," 6th Ed., 1952, p. 226) diluted 1:10, then left for 45 min. at room temp. before heating for 15 min. at 55°. For locating the spots, the chromatogram is immersed in *o*-tolidine - KI reagent (Reindel and Hoppe, *Chem. Ber.*, 1954, **87**, 1103). Experiments showed that procedure (*a*) located only amino-compounds, (*b*) located both amino- and imino-compounds, and (*c*) was the most sensitive for detection of the anomeric phenyl 2-acetamido-2-deoxy-D-glucosides.
D. C. ARMSWORTH

2223. **Glass apparatus for gas chromatography.** K. Otto and M. Doubek (Res. Inst. Macromol. Chem., Brno, Czechoslovakia). *Chem. Průmysl*, 1960, **10** (9), 476–477.—The main part of the apparatus is constructed from glass. The serpent-shaped column, thermal-conductivity cell and the sampling device are connected to form a unit. The apparatus was used for the analysis of mixtures of siloxanes, for the control of the quality of fluorinated hydrocarbons, and for the separation of unsaturated hydrocarbons, with samples in the 5 to 10-μl range. The thermal-conductivity detector and sampling device were used for the measurement of adsorption heats and for the determination of the surface areas of catalysts. J. ZÝKA

2224. **Technique for pyrolysing or vaporising samples for gas-chromatographic analysis.** H. Szymanski, C. Salinas and P. Kwitowski (Canisius Coll., Buffalo, N.Y., U.S.A.). *Nature*, 1960, **188**, 403–404.—Rapid heating by induction is used to prepare samples for gas chromatography. High temperatures are obtained and the application of this technique to the pyrolysis of polymers is described. Plaskon metal, as used in the induction heating of steel, is first mixed with the polymer and pyrolysis takes place with or without the carrier gas flowing over the sample. Suggested applications are listed. C. B. BAINES

2225. **[Switch-over mechanism for] sampl devices [for chromatographic analysis apparat** Perkin-Elmer Corp. Brit. Pat. 855,234, date ap 12.2.57. U.S.A., date appl. 27.2.56.—The swit over mechanism, which enables a part of the fl path of a stream of sample substance to be int duced into a stream of a carrier gas, has a connect conduit, by which the stream of sample substa is enclosed in the switched position. It has a dev which is connected to inlet and outlet conduits the sample to be analysed, being connected to in and outlet tubes for the carrier gas and affordin flow path either between the conduits or betwe the tubes and, in switching from one path to t other, encloses and transfers a sample.
J. M. JACOBS

2226. **Programmed-temperature gas chroma graphy.** Shun Araki, Ken Kishimoto and Yoshika Yasumori (Fac. of Engng, Tokyo Metropolit Univ., Fukazawa-cho, Setagaya-ku). *Jap Analyst*, 1959, **8** (11), 699–703.—A column of spi type is fixed on a heating plate; the temp. is rais electrically and controlled by a thermistor detect For decreasing the base-line drift (< 50 μV p 40 min.) caused by the rise of temp. of the colum the thermal-conductivity cell is kept in a thermos at a higher temp. than the highest programm temp. The flow rate of the reference side is restrict by inserting a packed column similar to the ch matographic one. K. SAITO

2227. **Split-temperature column system for g chromatographic analyses.** P. Y. Feng and B. Krotoszynski (Phys. Res. Div., Armour R Foundn, Chicago, Ill., U.S.A.). *Nature*, 1960, **1** 311–312.—To improve speed and resolution, t or more different temp. regions in the same unit a employed. This gives the combined advantages a low-temp. system, a high temp. system, and temp.-programmed system. The apparatus consi of a steel enclosure of 7 sections individually heat by heating tapes and controlled by bimetal thermo-regulators. The sections are partly i sulated from each other by glass-wool packings, a partly connected by metal strips, giving a unifo temp. in a section and a reasonably smooth tem gradient between sections. The system is complet by a 10-ft. glass column, packed with Apiezon L firebrick, connected to a Burrell Kromotog-K *Procedure*—Portions of the column are kept initia at lower temp. so that there is adequate resoluti of low-boiling components, and the remainder the column is kept at the higher final tem After the low-boiling components are eluted, t whole column is brought to the final temp. f analysis of the high-boiling substances. This spl temp. column system permits a resolution of t low-boiling components that compares favourab with that obtained with a low-temp. column, a a resolution of high-boiling components that co pares favourably with that obtained with a hig temp. column. The system has the advantage requiring only a short time to give satisfacto resolution. D. C. ARMSWORTH

2228. **Use of mixed stationary phases in ga liquid chromatography.** M. Šingliar, A. Bob J. Brída and L. Lukačovič (Res. Inst. of Pet Chem., Nováky, Czechoslovakia). *Z. anal. Che* 1960, **177** (3), 161–166 (in German).—The use polyoxyethylene glycol, diglycerol and diethyle glycol dibenzoate as stationary phases for t

tion of a mixture of iso- and n-butyr- ydes, toluene, n-butanol and isobutyl alcohol ustrated. The results indicate that good tions can be achieved with a column con- g a homogeneous mixture (33:67) of polyoxy- ne glycol and diglycerol.

B. B. Bauminger

9. **Calibration of the argon detector for volatile its.** R. R. Scott and B. W. Stannard (Bexford Brantham, nr. Manningtree, Essex, England). *& Ind.*, 1960, (41), 1259–1260.—Accurate ons of dichloromethane (**I**) in dry air are ned by passing the air at 300 cu. cm per min. gh **I** at −78° in an ether - solid CO_2 freezing ire. The concn. of the **I** in the mixture is mined by weighing the saturator before and the passage of air for 4 to 5 hr. (wt. loss 100 0 mg). Samples are introduced into the gas natograph via a 0·1-ml gas-sampling valve. concn. so calculated agrees fairly well with obtained by extrapolation of existing vapour- ure data. Calibrations of other volatile liquids nade by preparing a known liquid mixture ining **I** and injecting a few drops through um cap into a Winchester bottle, and, after ibration, withdrawing a sample of the gas to a natograph, measuring the peak heights and paring with the graph of **I** alone.

E. J. H. Birch

30. **A photo-ionisation detector for gases and urs.** J. E. Lovelock (Nat. Inst. for Med. Res., Hill, London, N.W.7). *Nature*, 1960, **188**, —The construction of a practical ionisation tor is described. An electrical discharge in f the rare gases or in N or H generates photons ergy sufficient for the ionisation of almost all atomic compounds. Stable discharges are ned at 100 torr or less with H, N or Ar, and tmospheric pressure with He. The detector a background current of 10^{-10} amp. and in the nce of ionisable vapour a substantially linear nse to vapour concn. is observed up to a signal nt of 10^{-7} amp. The response is unaffected by contamination of the detector surfaces and is e over long periods. C. B. Baines

31. **Accuracy of the determination of the position of mixtures with different methods of pretation of [gas] chromatograms.** N. M. el'taub, V. T. Shemyatenkova, N. A. Palamar- and L. A. Nechaeva. *Zavod. Lab.*, 1960, **26** 1075–1080.—Various methods of calculating ents from gas chromatograms are discussed. errors are least when it is possible to calculate roduct of the retention vol. and the peak height, use the peak height with allowance for the lardisation coeff. G. S. Smith

32. **Determination of trace impurities in gases as chromatography.** T. B. Kent (Instrument elopment Sect., I.C.I. Billingham Div., Co. nam). *Chem. & Ind.*, 1960, (41), 1260–1261.— centration techniques are not applicable to matic plant stream chromatographs for deter- ng trace impurities. It is preferable to inject mple directly into the column and use an argon ation detector capable of giving a good response changes of composition of 0·1 p.p.m. The mples of separation given are of 5 p.p.m. of ylene and 100 p.p.m. of propylene in N on a . column, of 5 p.p.m. of acetylene in ethane on 0-ft. column, and of 100 p.p.m. of acetylene in propene on an 80-ft. column. The stationary phase in all cases is 30% of dimethylsulpholane on 30 to 40-mesh brick at 20°. E. J. H. Birch

2233. **The analysis of dilute aqueous solutions by gas chromatography.** P. A. T. Swoboda (Low Temp. Res. Sta., Cambridge, England). *Chem. & Ind.*, 1960, (41), 1262–1263.—If an aq. soln. of alcohols is chromatographed on a stationary phase of a hydrophilic substance such as diglycerol, alcohols up to n-octanol emerge before the water, but are not well differentiated. This difficulty is overcome by arranging a side-arm in a 4-ft. gas-chromatography column which is packed before the side-arm with 6 in. of 20% diglycerol on Celite and beyond the side-arm with 40 in. of 10% polyoxyethylene glycol 400 on Celite. The 0·1% aq. soln. of alcohols (5 μl) is injected and the argon stream is allowed to pass through the whole column until the water is about to emerge from the diglycerol portion (2 min. 50 sec. at 30 ml of Ar per min. at 75°). The stream of Ar is then diverted to the side-arm when a portion chromatographs the alcohols on the polyoxyethylene glycol column, and a portion controlled by a capillary leak back-flushes the diglycerol column to atmosphere, thus preparing it for the next run. The alcohols are detected by an argon ionisation detector. The method could be used for any compounds eluted before water from a diglycerol column. E. J. H. Birch

2234. **Apparatus for continuous change of eluting agent for cation-exchange resin.** Giichi Muto, Masato Mamiya and Toshioki Amemiya (Inst. Ind. Sci., Tokyo Univ., Yayoi-cho, Chiba). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1959, **62** (5), 626–628. —The ion-exchange separation of binary mixtures (*e.g.*, Cu and Fe^{III}, Al and Fe^{III}, Mg and Ca, and Cu and Zn) was attempted with a new apparatus which permits the continuous change of the eluent, either by dilution of an acid of a known concn. or by the electrolysis of Na_2SO_4 (*e.g.*, at 5 V, 0·1 amp. per sq. cm). The former procedure is carried out by adding the acid to a large amount of water at a constant rate, which is greater than the rate of flow in the column. The separation is better than that achieved by step-wise change of the eluent.

K. Saito

See also Abstracts—**1819**, Symposium on gas chromatography. **2083**, **2084**, $SiCl_4$-treated paper for phosphatides.

Optical

2235. **Influence of goniometric arrangement and absorption in qualitative and quantitative analysis of powders by X-ray diffractometry.** J. Leroux and M. Mahmud (Occupational Health Div., Dept. of Nat. Health and Welfare, Ottawa, Canada). *Appl. Spectroscopy*, 1960, **14** (5), 131–134.—A general equation is deduced, and confirmed by experiment, and experimental conditions are suggested for analysis when goniometric arrangement and absorption by the sample have to be taken into account. A new type of grid-type sample-holder is suggested for attaining greater accuracy in the measurement of lattice spacings. P. T. Beale

2236. **Improvements in or relating to spectrographic apparatus.** E. Leitz G.m.b.H. Brit. Pat. 850,474; date appl. 16.7.57. Germany, date appl. 26.7.56.—Spectrographic apparatus for examining

(biological) objects, which exhibit stronger absorption in the non-visible spectral range than in the visible range, comprises a microscope suitable for creating an image of the object at an entrance slit of a spectrograph, a phase-contrast microscope having its optical axis inclined to that of the spectrograph, and a reflecting device, *e.g.*, a prism placed in the optical axis of the spectrograph, arranged to deflect visible light rays emanating from the slit in a lateral direction into the phase-contrast microscope, so that the latter can be used for observing the image and focusing it at the slit when visible light is used for illuminating the object. The apparatus is so arranged that the image, having been focused in visible light, can be recorded by the spectrograph in non-visible light J. M. JACOBS

2237. **Upper-scale expansion in infra-red absorptiometry.** B. S. Crawford, R. E. Dodd and D. G. Murchison (Dept. of Chem. and Geol., King's College, Newcastle upon Tyne, England). *Chem. & Ind.*, 1960, (39), 1210.—A third comb with triangular teeth has been used with a Grubb-Parsons double-beam i.r. spectrometer to enable the recorder scale to display the range from any chosen value to 100% extinction. The instrument is first balanced at zero with neither sample nor blank, secondly, the sample-beam trimming-comb is adjusted until the recorder indicates the value of extinction chosen as the new base line, and thirdly, the new comb is introduced into the reference beam until the balance position is again at zero; the sample and blank are then inserted. The sample-beam trimming-comb is then returned to its original position and the recorder zero will then be at the value of extinction chosen in the second step. For "base-lines" from 0 to 40% extinction, the measured extinction of a sample at 1600 cm^{-1} varied from 74 to 73%. R. M. S. HALL

2238. **Reduction of current fluctuations in the Farrand spectrofluorimeter.** D. J. Mahler, F. L. Humoller, H. G. Beenken and R. D. Loch (Med. Res. Lab., Veterans Admin. Hosp., Omaha 5, Nebr., U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1374.—By the modifications described, meter-needle fluctuations of the original instrument ranging from 20 to 30% of full scale were reduced to 1 to 2% of full scale. K. A. PROCTOR

2239. **An automatic hydrogen fluoride recorder proposed for industrial hygiene and stock monitoring.** D. F. Adams (Div. of Ind. Res., Washington State Univ., Pullman, U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1312–1316.—In the method described, concn. of F^- in the range 0·1 to 200 mg per cubic metre are determined by measuring the colour change produced by the reaction between F^- and the complex of 8-hydroxy-7-iodoquinoline-5-sulphonic acid with Fe^{3+}, with an automatic recorder. K. A. PROCTOR

2240. **Improvements in and relating to refractometers.** Bellingham and Stanley, Ltd. [Inventors: R. F. Stanley and J. Evans]. Brit. Pat. 848,243; date appl. 28.10.57.—The refractometer comprises a Littrow prism formed of, or adapted to contain, the medium of which the refractive index is to be measured; means, *e.g.*, a spherical mirror, for projecting a parallel beam of radiation on to the prism; means for rotating the prism to adjust the direction of the beam of radiation leaving the mirror; and means, *e.g.*, a reflector, for diverting part of the returning beam of radiation measuring and/or recording device. J. M. JAC

See also Abstract—**1990**, Non-dispersive photometer.

Electrical

2241. **Improvements in or relating to the mination of oxygen by electrical methods.** B Oxygen Research and Development, Ltd. ventor: J. H. Glover]. Brit. Pat. 851,512; appl. 29.5.58.—The concn. of oxygen in sol an electrolyte is determined by charging a pa electrodes in contact with the electrolyte predetermined potential, interrupting the cha *e.g.*, by breaking the circuit, and measurin drop in potential of the negatively charged elect which is proportional to the concn. of oxyg soln. in the electrolyte, after a predetermined interval. Concn. of oxygen in water or bloo other biological soln. over the range 0·02 to p.p.m. can be measured by this method. method can also be applied to the measure of the oxygen concn. in a gas by passing th through a suitable oxygen-free electrolyte, *e.g* KCl, until equilibrium is reached. By sui adjustment of the electrical circuit, concn oxygen over the range 0·005 to 100% by vol be measured. J. M. JAC

2242. **Constant cathode potential electro-depos apparatus.** N. J. Wadsworth (Metall. and Dept., Royal Aircraft Estab., Farnborough, H England). *Analyst*, 1960, **85**, 673–680.—An matic self-contained cathode potential ele deposition apparatus is described in detail. potential between a reference electrode and cathode is maintained within $\pm$ 5 mV of any set value in the range + 3 to − 3 V with cur up to 8 amp. Transistors are used throughou A. O. JON

2243. **Thermal electroanalysis. Effect of electroactive ions.** H. F. Holmes and M. J. Jo (Dept. of Chem., Univ. of Tennessee, Knox U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1251–12 The effect of non-electroactive ions on heat e at electrodes during thermal electroanalysi related to the thermodynamics of irrever processes as applied to non-isothermal, ele chemical cells. K. A. PROCT

2244. **Detector of the state of the cher constitution of a liquid.** Paterson Engineering Ltd, and A. C. Harvey. Brit. Pat. 848,587; appl. 30.4.58.—The detector makes use of measurement of an electric current prod as a result of depolarisation of a galvanic co formed by electrodes of dissimilar metals, plac the liquid. The electrodes are concentric annular in form, and are mounted in a rotatable for movement in contact with a bed of abra material for cleaning them. J. M. JACO

2245. **Techniques of a.c. polarography.** W Head, jun. (Eli Lilly and Co., Indianapolis, U.S.A.). *Anal. Chim. Acta*, 1960, **23** (3), 298 (in English).—Modifications to the Sar Model XXI polarograph for use with a.c. described. The a.c. converter is in an external and can be detached at will. While there i need to remove O it has been found that cer

give increased peak heights in the absence of g., Zn^{2+}, Pb^{2+}, Cd^{2+}, Fe^{2+}, Cr^{3+} and Ni^{2+}. organic compounds behave similarly. The ıce of the dropping electrode from the Hg electrode is of importance, and a means of ning improved damping is given. H. M.

46. **Improvements relating to polarography.** shed & Vignoles, Ltd. [Inventor: L. F. Nash]. Pat. 850,078; date appl. 13.2.57.—Apparatus ecording variations of current, or rate of ge of current, in a polarographic cell, with a ping mercury cathode, comprises a recording moving in relation to a chart surface under influence of damping to produce a trace senting the required polarogram. During the ds when the current in the polarographic cell anging rapidly, owing to the falling of one of mercury and the growth of the next, the is eliminated by means for inhibiting the ment of the recording point, which is operated ıe rapid change of current. A timing device so started to permit continued production of race after a predetermined time.

J. M. Jacobs

47. **Controlled-potential polarographic polari- unit with electronic scan and linear residual nt compensation.** M. T. Kelley, D. J. Fisher H. C. Jones (Anal. Chem. Div., Oak Ridge Lab., Tenn., U.S.A.). *Anal. Chem.*, 1960, 0), 1262–1265.—The unit described is particu- useful for both normal and derivative polaro- ıy at high sensitivity. Its performance is r than the conventional motor-driven multi- potentiometer scanner. K. A. Proctor

48. **Derivative polarography with solid rotating rodes in molten salts.** I. D. Panchenko (Inst. eneral and Inorg. Chem., Acad. Sci., Kiev). . *Anal. Khim.*, 1960, **15** (4), 388–390.—Deriva- polarograms are obtained for the successive arge of AgCl, $PbCl_2$ and $CdCl_2$ in a eutectic Cl and KCl as the supporting electrolyte. The ovský polarographic system is used with a ential circuit; the temp. is maintained at ± 2°, the galvanometer sensitivity is 1·6 $^{-9}$ amp. per mm, the electrolytic cells are lain dishes of 100 ml capacity, the cathode platinum wire (diam. 1 mm, length 8 mm) the anode a platinum plate (surface area . cm.). The decomposition voltage of the orting electrolyte is − 2·3 V, the $E_{\frac{1}{2}}$ is − 0·9 V ıg and − 0·95 V for Pb, and the first $E_{\frac{1}{2}}$ for s −1·1 V and the second is −1·3 V. The ing diffusion currents at concn. of 0·001M 0·24 mA for Ag, 0·32 mA for Pb, 0·16 mA he first Cd wave and 0·06 mA for the second ave; the value of the limiting diffusion current portional to the concn. of the substance being ed. It is shown that, with a solid electrode ing at 100, at 500 to 750 and at 1500 r.p.m., ncreases in the values of the diffusion currents wo-, three- and four-fold, respectively.

K. R. Cook

49. **Sulphonated phenylstearic acid as a polaro- ic maxima suppressor.** W. U. Malik and R. ıe (Dept. of Chem., Muslim Univ., Aligarh,). *Anal. Chem.*, 1960, **32** (11), 1528–1529.— use of sulphonated phenylstearic acid is shown ıve advantages over normal maxima suppres- Although an anionic soap, it can suppress both positive maxima (*e.g.*, of Pb^{2+}, copper - biuret complex, copper - glycine complex and cystine) and negative maxima (*e.g.*, of CdI_4^{2-}, Co^{2+}, Ni^{2+} and Co^{2+} - Ni^{2+}). C. B. Baines

2250. **Measuring assemblies for the determination of ion concentrations and redox potentials, particularly suitable for carrying out measurements at elevated temperatures.** W. Ingold. Brit. Pat. 850,177; date appl. 15.3.57. Switzerland, date appl. 16.3.56.—A lead-off system for high-temp. reference electrodes comprises a silver - silver chloride half-cell in which a silver wire is surrounded by and in contact with solid, discrete particles of silver chloride (provided with a thin coating of metallic silver) so that the silver chloride concn. is held at a level corresponding to the working temp., thus ensuring that the potential of the half-cell is held at a constant value for any specified temp. in the range from 70° to ⩽ 130°. The assemblies are particularly suitable for determining the pH of nutrient broths for the production of antibiotics. J. M. Jacobs

2251. **Acid - base titrations with the tellurium electrode.** J. A. Ricketts and R. E. Bowen (DePauw Univ., Greencastle, Ind., U.S.A.). *J. Chem. Educ.*, 1960, **37** (9), 473–475.—The reversible oxidation potential of the TeO_2 - Te^{4+} electrode is linear with respect to pH between 0 and 9 at 20° to 25°. For acid - base titrimetry a platinum-wire or -foil electrode freshly plated with amorphous tellurium (from 5% aq. Na_2CO_3 soln. satd. with TeO_2) is preferred. The titration is made potentiometrically with the Te - S.C.E. couple, the p.d. of which is not measured until 2 min. after the addition of each increment of titrant. By simultaneous use during the titration of a glass electrode and pH meter, a comparison of the Te and glass electrodes in acid - base titrimetry can be made.

W. J. Baker

2252. **The glass electrode as indicator electrode for potentiometric titrations in non-aqueous solutions.** H. Frind and A. Busch (Cassella Farbwerke Mainkur A.-G., Frankfurt/Main, Germany). *ChemikerZtg*, 1960, **84** (17), 568–569.—In this review 19 references (17 from *Anal. Chem.*) concerned with titrations of numerous org. and inorg. compounds in non-aqueous media are presented in tabular form. E. G. Cummins

2253. **Acidimetric and iodimetric determinations with the high-frequency titrimeter.** E. Pungor and L. Balázs (Inst. f. anal. Chem., L. Eötvös Univ., Budapest, Hungary). *Mikrochim. Acta*, 1960, (5–6), 678–684 (in German).—A titrimeter working at 130 MHz is used for the determination with 0·1N-aq. NH_3 of oxalic, malonic, tartaric and citric acid and of CO_3^{2-} and HCO_3^- in the presence of each other with 0·1N-HCl or acetic acid, and 0·1N-NaOH. It is also used for the iodimetry of SO_3^{2-}, As^{3+}, and Sb^{3+} with 0·1N-iodine in methanol.

J. P. Stern

2254. **Alternating-current voltammetry at solid electrodes.** D. E. Walker, R. N. Adams and A. L. Juliard (Dept. of Chem., Univ. of Kansas, Lawrence, U.S.A.). *Anal. Chem.*, 1960, **32** (11), 1526–1528.—Alterations to a normal voltage-scan instrument are described for the construction of an instrument suitable for a.c. voltammetry at solid electrodes. The electrode system consists of

a polarisable electrode and a S.C.E. in the d.c. branch, and a polarisable electrode and platinum foil in the form of a cylinder in the a.c. branch. A platinum wire, a carbon-paste electrode and a graphite rod have all been used successfully as polarisable electrodes for a.c. voltammetry. The technique shows promise for the study of the oxidation of many organic compounds, facilitates the study of electrode mechanisms, and may be used in non-aqueous media. C. B. Baines

2255. **The graphite electrode: an improved technique for voltammetry and chronopotentiometry.** P. J. Elving and D. L. Smith (Univ., Ann Arbor, Mich., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. TID–6167, 1960. 39 pp.—The application of the graphite electrode to voltammetry, which has been improved by wax impregnation of the electrode, by insulation, and by surface renewal by turning, was further improved by pre-wetting of the electrode surface by a dil. soln. of a wetting agent. The performance of the electrode so prepared and conditioned was evaluated for quant. analysis by cathodic and anodic chronopotentiometric and voltammetric measurements in aq. soln. on the ferricyanide - ferrocyande and quinone - quinol systems, and on adenine, ascorbic acid, sulphanilamide, and tetraphenylborate. Chronopotentiometric transition time constants were within $\pm$ 3%; a single calibration plot of $i\tau^{\frac{1}{2}}$ *vs.* concn. suffices for quant. work. Comparable results are obtained by automatic recording voltammetry. The results compare favourably with those obtained with platinum electrodes.

Nucl. Sci. Abstr.

2256. **Coulometric passage of reagents through ion-exchange membranes.** R. B. Hanselman and L. B. Rogers (Dept. of Chem. and Lab. for Nucl. Sci., M.I.T., Cambridge, Mass., U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1240–1245.—The passage of ions through selective permeable membranes enlarges the number of reagents available for coulometric titrations. Chloride, Br^-, OH^-, Ag^+, Ca^{2+} and hydronium ions have been generated at a constant current with efficiencies between 90 and 105% and standard deviations better than 2%. Variables that will be important in extending the technique to other reagents have been evaluated.

K. A. Proctor

2257. **Automatic coulometric titrator. Application to the determination of sulphur in petroleum by high-frequency combustion.** J. R. Glass and E. J. Moore (Res. Dept., Socony Mobil Oil Co., Inc., Paulsboro, N.J., U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1265–1269.—In the method described, sulphur is oxidised to SO_2 which is titrated as it is formed by electrically generated iodine. The instrument is applicable to acid - base, precipitation, and oxidation - reduction titrations.

K. A. Proctor

2258. **A coulometric borane monitor.** R. S. Braman, D. D. DeFord, T. N. Johnston and L. J. Kuhns (Callery Chemical Co., Pa,. U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1258–1262.—In the apparatus described, boranes, scrubbed out of air into a $NaHCO_3$ - KI electrolyte, are titrated with coulometrically generated iodine. Diborane and decaborane in concn. as low as 0·2 p.p.m. can be determined, but the various boranes cannot be differentiated. Materials that react with iod such as acetone and peroxides, interfere.

K. A. Procto

2259. **Method for analysing gaseous or liq mixtures.** H. Hummel. Brit. Pat. 850,044, d appl. 16.7.58. Germany, date appl. 18.7.57.- fluid medium, especially a gaseous medium, analysed by measuring its dielectric constant. medium is introduced into a chamber forn between the plates of a capacitor, or into a cav resonator, and then submitted to periodic chan of pressure. The resulting periodic changes the electrical characteristics of the capacitor used to provide a measure for determining composition of the medium being tested or concn. of one of its constituents. J. M. Jacob

2260. **Instrument for measuring the activity radio-isotopes.** K. Subrahmanyam (Electronics D Atomic Energy Establ., Trombay, Bombay). *Sci. Ind. Res., India, A*, 1960, **19** (5), 212–215 The instrument described can measure activit from 10 μC to several curies of radium-equival as a γ-emitter and from 0·5 μC upwards of ^{32}P a β-emitter. Details of the ionisation cham are given and the circuitry is designed in such way that the activity of the source is read direc on a meter calibrated in curies. G. Skirrov

2261. **Quenching of fluorescence in liquid scin lation counting of labelled organic compoun** C. T. Peng (School of Pharm., Univ. of Califor Med. Centre, San Francisco, U.S.A.). *Anal. Che* 1960, **32** (10), 1292–1296.—A method of correct for fluorescence quenching without the use of internal standard is reported. The quench constant or half-quenching concn. varied w instrumental amplification of the pulse signal a particle energy. K. A. Procto

2262. **Backgrounds for liquid scintillation cou ing of coloured solutions.** R. J. Herberg (L Res. Lab., Indianapolis, Ind., U.S.A.). *An Chem.*, 1960, **32** (11), 1468–1471.—The meth described overcomes the difficulty of obtain background soln. of the same colour intensity those of the sample soln. The reciprocal exti tion at 400 mμ of coloured soln. is a linear funct of their relative counting efficiencies, and refere of measurements at this wavelength to stand curves is the basis of the correction procedure o lined. Isotope and background constants for and ^3H in blood digest soln., and methyl red a bromothymol blue systems are given.

C. B. Baine

2263. **Further development of the two-stage m spectrometer for isotopic analysis of uraniu** L. A. Dietz, C. F. Pachucki, J. C. Sheffield, A. Hance and L. R. Hanrahan (Knolls Atomic Po Lab., General Electric Co., Schenectady, N U.S.A.). *Anal. Chem.*, 1960, **32** (10), 1276–1278 Developments of the two-stage mass spectrom designed by White and Collins (*Appl. Spectrosco* 1954, **8**, 169) are described. Voltage discriminat for the isotopic ratio $^{238}U^+$ to $^{235}U^+$ is $\simeq$ 0·1% 15-kV ions. K. A. Procto

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See also Abstracts—**1820**, Proceedings of radioa vation symposium. **1821**, Reagent addition a potential change. **1870**, A.c. polarisation titrati

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	A
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
Colour Index	C.I.
concentrated	conc.
concentration	concn.
constant	const.
crystalline	cryst.
crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
equivalent	equiv.
ethylenediaminetetra-acetic acid	EDTA
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_l
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

The chemical nomenclature used in the abstracts is that recommended by The Chemical Society ("Handbook for Chemical Society Authors", The Chemical Society, London, 1960), and may differ from that used in the original articles.

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